

CHAPTER — V

SUMMARY

S U M M A R Y

Considerable work has been done on electroluminescence (El.), since the time of Destriau ( 1937 ). Current literature provides an empirical and descriptive account of electroluminescence in various forms, in which the phenomenon has been observed on different materials. The best known El.phosphors have a zinc sulphide matrix. The first report of El.in organic crystal is probably attributable to Pope et al. who performed measurements on single crystals of anthracene and anthracene/doped with tetracene.

From the survey of literature, it seems that no systematic efforts have been made to investigate the electroluminescent properties of metallic salts of 8-hydroxyquinoline. The investigation of organic El. often proves to be quite exciting since it combines a number of research areas such as electronics, spectroscopy, electro-chemistry and photochemistry, both in solid state and in solution state.

PURIFICATION OF 8 - HYDROXYQUINOLINE ANDPREPARATION OF METALLIC SALTS OF8 - HYDROXYQUINOLINE.

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 amount 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. The BDH grade 8-hydroxyquinoline was repeatedly recrystallised in 75% absolute alcohol. The purity of oxine was confirmed by following recommended thin layer chromatography.

8-Hydroxyquinoline (oxine) forms sparingly soluble derivatives with metallic ions, which have the composition  $M (C_9 H_6 ON)_2$  if the co-ordination number of the metal is four,  $M (C_9 H_6 ON)_3$  if the co-ordination number is six and  $M (C_9 H_6 ON)_4$  if the co-ordination number is eight. The solid metal complexes of 8-hydroxyquinoline were prepared by the methods of Kolthoff and Sandell. The preparative methods for all the metallic salts are the same except, pH of the precipitating solution and drying temperatures.

#### M E T H O D O F T E S T I N G

The powdered salt of 8-hydroxyquinoline was sieved in and its pasty suspension in a alcohol (as well as various dielectrics) was applied on to a aluminium sheet. This was covered by a thin mica sheet and a transparent conducting glass at the top; suitable lead wires were taken out and the a.c. field was applied. The condensor-like cell assembly was enclosed in a suitable ebonite frame. The emitted light was observed through the conducting glass. Photoluminescence of these prepared compounds was tested visually by using short wavelength U.V. light (ULTRA-VIOLET PRODUCTS, INC. SAN GABRIEL, CALIF.).

## STUDY OF ELECTRO - OPTICAL PROPERTIES,

A set up consisting of following components was designed to study the electro-optical properties of metallic salts of 8- hydroxyquinoline .

- a) a sample holder for an El. cell,
- b) excitation unit- an audio-frequency oscillator (Philips, GM 2308, India) coupled with step-up transformer for voltage and frequency variation,
- c) dispersion unit- Carl Zeiss Jena ( Spekol) high intensity grating monochromator,
- d) detecting unit - a photomultiplier tube (IP-21) with a powerpack unit, and
- e) intensity measuring unit- a sensitive spot reflecting galvanometer (Polyflex, Toshniwal, sensitivity =  $1.2 \times 10^{-9}$  amps per mm deflection).

In order to obtain reproducibility, the excitation conditions like cell spacing, the area of illumination, applied voltage and frequency were kept under close control.

## ELECTROLUMINESCENCE SPECTRA AND

### MECHANISM OF ELECTROLUMINESCENCE,

The spectral intensity distribution of El. emission ( El - spectra ) of electroluminescent metallo-oxinates at various excitation voltages and frequencies were studied. For all the materials studied, the El-spectra

were found to be identical with their normal fluorescence spectra. It was also observed that  $\lambda_{\text{max}}$  in each case is independent of applied voltage and frequency of excitation, over entire range studied.

A general trend of decreasing intensity with increasing atomic number was observed in the studied complexes. In the discussion of El.-spectra, the broad considerations for the important process affecting the luminescence of metal chelates were considered, namely,

- a) the nature of the ligand,
  - b) the nature of the metal,
  - c) the nature of the perturbations,
- and d) intramolecular energy transfer processes.

Our experimental observations are exactly identical with the previously reported electroluminescence in various fluorescent organic compounds, the results of which were explained on the basis of glow discharge mechanism. The metallic oxinates of Mg, Ca, Zn, Cd and Al are found to be highly photoluminescent, while that of Mn, Cr, Fe, Co, Ni, Cu, Hg and Sn are non-photoluminescent. These compounds when tested for their El. behaviour it was found that only the photoluminescent oxinates show bright El. emission while the non-photoluminescent oxinates were non-electroluminescent. This factor strongly supports the validity of glow discharge mechanism of El. emission from our metallo-complexes of 8-hydroxyquinoline.

In glow discharge mechanism, when high electric field was applied between the two electrodes, breakdown of the gas surrounding the cell can occur, resulting in light emission from the gas and if a fluorescent material is present, the light emitted by the gas can cause photoexcitation of the material in the cell i.e. nitrogen molecules from the air gets excited and emit ultraviolet (U.V) radiations. These U.V. radiations excite the fluorescent material which latter on emits the normal fluorescence emission.

VOLTAGE AND FREQUENCY DEPENDENCE OF  
ELECTROLUMINESCENCE EMISSION,

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The general and some what surprising, effect of the glow emission mechanism is that the electrical properties exhibited are very similar to those observed for genuine inorganic El. which will be evident from our study on frequency and voltage dependence of El. emittance. For all the metal chelate complexes studied, it was invariably observed that for a constant high frequency, the El. brightness (B) increased exponentially with applied voltage V, and seems to saturate at high voltages. The various well established equations for B-V relation were tested and it was found that the equation -

$$B = a. \exp \left[ \frac{-b}{V} \right]$$

describing the saturation behaviour is obeyed for all

organo-metallic complexes. In the complexes studied, if there are no junctions or barriers,  $E$  (the electric field strength) would be proportional to  $V^{\frac{1}{2}}$ . Thus, it seems that electrons are introduced by the field, either from the electrodes or from bound states within the material, at a rate proportional to  $V^{\frac{1}{2}}$ . These electrons may excite luminescence centres in the material and thus light is produced.

At constant voltage, E.l. brightness increases with frequency and seems to saturate at higher frequencies. The results have been explained on the basis of electroluminescent build-up process.

The luminous efficiency, found to be depend<sup>ent</sup> upon the voltage and frequency of the applied field. For a given metallic-salt, the efficiency increases rapidly with voltage and frequency and reaches a saturation value. Thus, E.l. efficiency seems to be optimum at a specific voltage and frequency only.

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