CHAPTER 2

EXPERIMENTAL : MATERIALS, METHODS AND APPARATUS

2.1 Experimental

The general descriptions of the experimental techniques used in this research are given below. Details of a particular experimental method will be described in conjunction with the discussion of the results thereby obtained.

2.2 Chemicals

The polyene semiconductors used in this work are vitamin A alcohol (*all-trans*), vitamin A acetate (*all-trans*), β -apo-8'-carotenal, astacene and methyl bixin. Chemical structures of these polyenes have already been shown in chapter 2, part I. The organic liquids used in this investigation are toluene, benzene, ethyl acetate, n-heptane, ethanol and methanol. These liquids were of spectrograde or equivalent quality of B.D.H (England) and E. Merck (Germany). The teflon spacers and sheets, and the conducting glass electrodes required for this work were obtained from Dielectrix Corporation (U.S.A.) and Fisher Scientific Co. (U.S.A.) respectively.

2.3 Preparation of the " Sandwich " Conductivity Cell

Applying the usual procedure[1-4] the conductivity cells were made in air by placing about 5 mgs of the semiconductive materials on a clean stainless steel electrode (plate) in safe light illumination. Two teflon spacers, 2 mils (0.005 cm) thick, were positioned near the edges of the electrode, and the powdered crystals were flattened by gently rotating a piece of conducting glass electrode on the top with the conducting side making contact with the specimen. The teflon spacer maintained the separation between the electrodes. To maintain the sandwich cell, two spring clips were fixed at a moderate pressure to the ends of the electrodes.

2.4 Experimental Arrangements :

A schematic diagram of the experimental set-up for the studies of semiconductivity presented in Fig. 2.1 is similar to that of Rosenberg et al[2,5]. The sandwich cells were placed in a conductivity chamber made of brass and fashioned entirely with teflon - all the electrical surface leakage parts were of teflon. The stainless steel electrode was placed on a thermal copper bar platform in good thermal contact through thermal paste and thus the temperature of the sandwich cell could be controlled from outside. A d.c. voltage of 22.5 volts from dry batteries was applied across the cells. There was a gas inlet and an outlet in the chamber for gas adsorption study and an arrangement for connecting the outlet with a suction pump. The chamber atmosphere could circulate freely through the opposite open sides of the sandwich cell. Temperature measurements were made by using a copper-constantan thermocouple attached at the top of the metal electrode and a millivolt potentiometer of Toshniwal Brothers Pvt. Ltd., India. The semiconduction current measurements were made with an electrometer amplifier, EA 815, of the Electronic Corporation of India Ltd. In order to eliminate the effect of oxygen, water vapour or any other vapours or gases adsorbed by the sample before

FIG. 2.1 : A schematic diagram of the apparatus used to test the effects of the adsorbed vapours (or gases) on the conductivity of the polyene semiconductors.

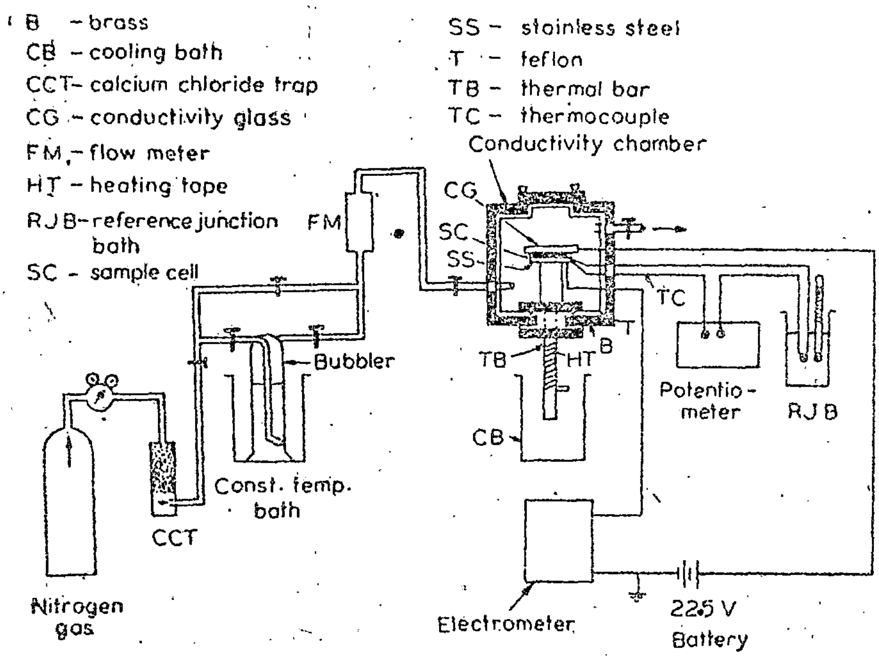
B - brass
CB - cooling bath
CCT- calcium chloride trap
CG - conductivity glass
FM - flow meter
HT - heating tape
RJB- reference junction bath
SC - sample cell
SS - stainless steel
T - teflon
TB - thermal bar
TC - thermocouple
Conductivity chamber
FM
CG
SC
SS
TC
T
B
TB
HT
Bubbler
CB
Potentio-meter
RJB
Const. temp. bath
CCT
Nitrogen gas
Electrometer
22.5 V Battery

FIG. 2·1

experiment, the chamber was thoroughly flushed with dry nitrogen gas and the sample was then given repeated heating and cooling treatments in nitrogen atmosphere over the temperature range to be used. To pass various vapours inside the chamber, dry nitrogen gas was used as a carrier which was passed through the bubbler containing the organic liquid kept at a required temperature to maintain a fixed partial vapour pressure less than the saturation vapour pressure at sample cell temperatures. For desorption studies, dry nitrogen gas was allowed to pass directly through the chamber. The measurements of dark currents at a constant or different cell temperatures were carried out maintaining the conductivity cells at nitrogen, vacuum or different ambient atmospheres according to our experimental requirement.

References

1. T.N. Misra, B. Rosenberg and R. Switzer, J. Chem. Phys.,
48, 2096 (1968).

2. B. Rosenberg and H.C. Harder, Photochemistry and Photobiology,
6, 629 (1967).

3. B. Rosenberg, J. Chem. Phys., 34, 63 (1961).

4. B. Rosenberg, J. Chem. Phys., 31, 238 (1959).

5. B. Rosenberg, T.N. Misra and R. Switzer, Nature, 217,
423 (1968).