CHAPTER 2

MATERIALS AND INSTRUMENTAL TECHNIQUES USED

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2.1 Chemicals and solvents:

All chemicals used were of the reagent grade quality and used without further purification. The solvents were dried before use following standard procedures. The starting materials were procured from Tokyo Kasei (Japan), Lancaster Chemicals (USA), Sigma Aldrich (USA) and TCI Chemicals (Japan). Chromatographic separation were carried out using Silica (60-120 mesh) from Spectrochem. Silica gel G (E-Merck, India) was used for thin layer chromatography (TLC).

2.2 Instrumental techniques used:

CHN analysis: The C, H and N analyses were carried out using Carlo Erba 1108 elemental analyzer.

Absorption Spectra: UV-visible spectra of the compounds in different solvents were recorded on Shimadzu UV-1601PC, JASCO V-670 and Perkin Elmer Lambda 35 spectrophotometers on different occasions. Spectra of the complexes were recorded at room temperature in the wavelength range 200-800 nm.

FT-IR Spectroscopy: Infrared spectra were recorded on a Perkin Elmer BX series spectrometer on KBr discs in the 400-4000 cm^{-1} range.

NMR spectroscopy: The ^{1}H and ^{13}C NMR spectra were recorded on Bruker Avance II 400 MHz spectrometer in $CDCl_3$ (chemical shift in δ) solution with Tetramethylsilane (TMS) as internal standard.

FAB mass analysis: Mass spectra were recorded on a JEOL SX-102 spectrometer with fast atom bombardment.

Photoluminescence study: Photoluminescence spectra of the compounds were recorded on Shimadzu RF-5301PC, Perkin Elmer LS 45 and Hitachi F-4600 Fluorescence spectrophotometers on different instances. The fluorescence quantum yield in dichloromethane was determined by optical dilution method using 9, 10-Diphenylanthracene (EQY = 0.96, in cyclohexane) as standard. Quantum yield in the solid state was measured by means of an integrating sphere, in which the solid sample film was prepared via spin coating and was excited by a pulsed xenon source. The resulting luminescence was acquired by an intensified charge-coupled detector for subsequent analyses.

Polarizing Optical Microscopy: The optical textures in the different phase of the compounds were studied by placing a small amount of the LC sample between a glass plate and a cover slip, kept in the path of white light beam crossed with polarizers using a Nikon ECLIPSE LV100 POL polarizing microscope attached with Instec hot and cold stage HCS402 with a STC200 temperature controller of 0.1 °C accuracy. Since the sample is birefringent, interference colours appeared which resulted in beautiful textures characteristic of molecular arrangement. The phase transition temperatures were detected and associated textures of different liquid crystalline phases were observed. The textures at different temperatures were recorded using photo micrographic equipment attached with the polarizing microscope. The liquid crystalline properties were established by thermal microscopy and the phase transitions were confirmed by differential scanning calorimetry.

Differential Scanning Calorimetry: The thermal behaviour of the compounds was studied using a Pyris-1 system linked to a Perkin Elmer differential scanning calorimeter (DSC) at a heating or cooling rate of 5 °C min^{-1}.

PXRD study: Variable temperature powder X-ray diffraction (PXRD) experiments were performed in the transmission geometry with the samples in a glass capillary (Capillary

Tube Supplies Ltd, UK). The XRD apparatus (X'Pert PRO MP, PANalytical), employing Cu Kα (l = 0.15418 nm) radiation, consisted of a focusing elliptical mirror for beam preparation optics providing a well-focused line beam, a fast high resolution multi-channel solid state detector (PIXCEL) and operated at 45 kV and 30 mA rating. Collimation was carried out with 20 mrad Soller slits on the input as well as the diffracted beam side providing very good vertical resolution.

DFT study: Quantum chemical calculation on some selected compounds was performed using density functional theory (DFT) as implemented in DMol3/GAUSSIAN 09 package at BLYP/B3LYP level.