

Part II

Thermoanalytical studies

Chapter 2

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CHAPTER 2

MATERIALS, METHODS AND INSTRUMENTS

Reagents

Analar grade chemicals supplied by E. Merck were used for the synthetic strategies of the compounds. Commercial solvents were distilled by adopting the standard methods. Detailed discussions regarding the reagents are given in Part I.

Preparation of ligands and complexes

The Schiff base ligands, 3-(1H-indol-3-yl)-2-[(E)-(thiophen-2-ylmethylidene)amino]propanoic acid (I3YT2YMAPA), (E)-3-[thiophen-2-ylmethyleneamino]benzoic acid (T2YMABA), (E)-4-(5-[(2-carbamothioylhydrazono)methyl]thiophen-2-yl)benzoic acid (CTHMT2YBA), (E)-4-(5-[(2-phenylhydrazono)methyl]thiophen-2-yl)benzoic acid (PHMT2YBA) and (E)-4-(5-[(2-carbamothioylhydrazono)methyl]furan-2-yl)benzoic acid (CTHMF2YBA) were synthesized by the condensation reaction between the corresponding carbonyl compound and the amino compound in ethanolic medium. The Cr(III) and Ni(II) complexes of these ligands were prepared by refluxing with the corresponding metal acetates. The preparative aspects of these ligands and complexes are explained in Part I.

Instruments

The instruments used for the present thermoanalytical studies are Perkin Elmer STA 6000/Diamond TG/DTA thermal analyser system with PT-PT/Rh (Type R) thermocouples and Bruker AXS D8 advance diffractometer.

Thermogravimetric analyses of the coordination complexes were carried out with air as the initial purge gas, at a heating rate of $10^{\circ}\text{C}/\text{min}$ employing about 3-7mg of each complex.