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# physico chemical studies on transition metal chelates with Schiff bases derived from dimine with acetylacetophenol

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In the first chapter, a literature survey of the previous studies on Schiff bases and their complexes with different metal ions is given. This survey includes; potentiometric, spectrophotometric and studies on Schiff bases and their solid complexes. It includes also the use of some Schiff bases as analytical reagents for detection and determination of transition metal ions spectrophotometrically. The experimental part (chapter-II) includes the preparation of the Schiff bases under investigation. It comprises also information about the instruments used for conductometric, potentiometric, spectrophotometric, IR as well as <sup>1</sup>H NMR. Chapter III includes the results and discussion and consists of two parts, the first part (A), comprising the studies in solutions of complexes formed between Schiff bases under investigation with transition metal ions (Cr<sup>3+</sup>, Fe<sup>3+</sup>, Co<sup>2+</sup>, Ni<sup>2+</sup>, Cu<sup>2+</sup> and Cd<sup>2+</sup>). The studies include conductometric, potentiometric (at different temperatures; 298, 308 and 318 K) and spectrophotometric techniques from the conductometric measurements the stoichiometry of the different complexes is obtained. Potentiometric titrations of Schiff bases together with Cr<sup>3+</sup>, Fe<sup>3+</sup>, Co<sup>2+</sup>, Ni<sup>2+</sup>, Cu<sup>2+</sup> and Cd<sup>2+</sup> ions are performed in a medium of hydrochloric acid and potassium chloride. The proton-ligand formation constants log K<sub>HL</sub> are determined as well as the formation constants of the complexes log K<sub>M</sub>. The stoichiometry and stability constants of metal complexes are also evaluated from the spectrophotometric methods namely the molar ratio and continuous variation methods. The optimum conditions for the complex formation are investigated, universal buffer of 30% (v/v) ethanol was found to be the best medium for spectrophotometric studies. Optimum pH values, suitable wavelength, effect of time and temperature as well as the sequence of addition were also studied. The results obtained indicate that a satisfactory agreement is observed between stability constant values evaluated using spectrophotometry and potentiometry. Part (B) of chapter (III) includes studies of the solid chelates which include elemental analysis, molar conductivity measurements, IR and <sup>1</sup>H-NMR. The molar conductance of the complexes in DMF shows that these chelates are low electrolytes in nature and disassociate into ions. The stability constants were determined using the data obtained from potentiometric titration and elemental analysis. The IR-spectra of the metal chelates are studied and compared with those of the free ligands which indicate that coordinate and covalent bonds occur through the oxygen atoms of the carbonyl

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groups and the oxygen atoms of phenolic hydroxyl groups form and nitrogen atoms of the imino groups. The IR-spectra of the metal chelates exhibit a very broad band at high frequency due to the water molecules coordinated to the central metal ions. The  $^1\text{H-NMR}$  spectra of  $\text{Cd}^{2+}$  - chelates with the ligands under investigation were studied in DMSO and compared with those of the free ligands. The main signals due to the protons of the phenyl ring, CH, CH<sub>2</sub> and CH<sub>3</sub> groups are detected. It is found that the signals of protons of the NH groups of the diamine part completely disappeared in the  $\text{Cd}^{2+}$  -chelates indicating contribution of NH groups in chelation through proton displacement i.e.  $\text{Cd}^{2+}$  enter in an- N2O2 compartment. 4- Chapter (IV) includes the analytical microdetermination of transition metals ions under consideration  $\text{Cr}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Cu}^{2+}$ , and  $\text{Cd}^{2+}$  with Schiff bases under investigation using the spectrophotometric techniques (Calibration curve and EDTA titration). Analytical application for the microdetermination of vitamin B12 (Cyanocobal-amine) in pure and in pharmaceutical formulations are reported.