

SPECTRAL AND THERMAL STUDIES ON COPPER(II) COMPLEXES OF ACENAPHTHAQUINONEMONO-(4-METHYLQUINOLINYL)HYDRAZONE

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Cu(II) complexes of acenaphthaquinonemono(4-methylquinoliny)hydrazone (AMH) of general composition $[CuLX_2]$ (where $L = AMH$; $X = Cl, Br, I, OAc$ or NO_3) with the exception of sulphato complex, where the composition is found to be $[CuLSO_4]_2$ have been synthesized and characterized by elemental analyses, magnetic moment measurements, conductivity measurements, IR, electronic and EPR spectral techniques and by thermal analysis. A planar geometry is indicated for all the complexes. TG curves show one step decomposition of complexes and formation of Cu_2O at the end of the step.

Keywords: complexes, coordination chemistry

Introduction

Coordination chemistry of substituted hydrazones has been provided by remarkable anticancer [1], antibacterial [1], antileukemic [2] activity observed for these compounds which has since been shown to be related to their metal complexing ability. Substituted hydrazones exhibit a wide range of stereochemistries on complexation with metal ions [3-5]. In this paper we describe the synthesis, spectroscopic characterization and thermal studies on Cu(II) complexes of AMH.

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Experimental

Physical measurements

The complexes were analysed for C, H and N in Microanalytical Laboratory, I.I.T., New Delhi (India). Copper was estimated on a Perkin-Elmer 703 atomic absorption spectrophotometer in solutions prepared by decomposing the complexes in conc. HNO_3 . Magnetic susceptibility measurements at room temperature were carried out in the powdered state on a vibrating sample magnetometer PAR 155 with 5000G field strength, using high purity nickel metal (saturation moment 55 emu g^{-1}) as calibrant. Molar conductances of complexes in DMF solution were measured at room temperature on a systronic digital direct reading conductivity meter model 304 using platinum electrodes. The electronic spectra (900–300 nm) of the complexes in ethanol were recorded on a Perkin-Elmer 554 UV visible spectrometer. IR spectra were reported in the range 4000–400 and 800–200 cm^{-1} as KBr and CsI discs, respectively. EPR spectra in polycrystalline state at room temperature were recorded on a varian model E-4 spectrometer (X-band) using DPPH ($g = 2.0036$) as the standard. The PMR spectra of the ligand was recorded in CDCl_3 on Perkin-Elmer model R-32 NMR spectrometer operated at 90 MHz. TG was recorded on Stanton Red-Croft TG 770 thermobalance using 5–10 mg samples and the heating rate was $3 \text{ deg}\cdot\text{min}^{-1}$.

Materials and methods

All the reagents used were of commercial grade and were used without further purification.

Preparation of AMH

2-Chloro-4-methylquinoline synthesized by reported method [6–7] was refluxed with hydrazinehydrate which gives 2-hydrazino-4-methylquinoline. 2-Hydrazino-4-methylquinoline (3.46 g, 0.02 mol) was dissolved in 25 ml of HCl. Acenaphthaquinone (3.64 g, 0.02 mol) dissolved in 100 ml of ethanol was added to 4-methylquinolinylhydrazinehydrochloride. The contents were refluxed for half an hour and the product obtained was filtered, recrystallized from ethanol and dried in vacuum desiccator over P_2O_5 (Found: C, 78.68; H, 4.51; N, 12.43%. Calc. for $\text{C}_{12}\text{H}_{15}\text{N}_3\text{O}$: C, 78.33; H, 4.45; N, 12.46%).

The purity of the ligand was checked by TLC and characterized by elemental analysis, IR and PMR spectroscopy (δ value ppm) in CDCl_3 [2.4(s, 3H, -C- CH_3); 7.2–7.9(m, 9H, aromatic protons); 8.8(s, 1H, N-NH-C); 8.2(s, 1H, ring NH protons)].

Synthesis of complexes

Ethanolic-water solution of various Cu(II) salts (1 mmol) was added to an ethanolic solution of AMH (2 mmol). The resulting mixture was refluxed for 3h