

NOTES

IRON (II) COMPLEXES WITH TWO NEW BICHALCONES

G. HEMALATHA, A. JOHN MERINA, V.R. VENKATARAMAN and S. NAGARAJAN*

*Department of Chemistry
Bharathidasan University, Tiruchirapalli-620 024, India*

*Two new bichalcones 2',4'-dihydroxy-5'-(2-hydroxy) cinnamoyl-2-hydroxy-chalcone and 2',4'-dihydroxy-5'-(4-hydroxy-3-methoxy) cinnamoyl-4-hydroxy-3-methoxychalcone have been synthesised and characterised by IR and PMR studies. Their iron (II) complexes have been prepared and characterised by elemental analysis, IR, magnetic and conductivity measurements.

Bivalent metal complexes of 2'-hydroxy-chalcone and its substituted derivatives are widely reported in the literature^{1,2}. Studies on Ni(II) and Cu(II) complexes with a bichalcone have been recently made³. The present paper deal with characterisation of Fe(II) complexes of two new bichalcones.

2',4'-Dihydroxy-5'-(2-Hydroxy) Cinnamoyl-2-Hydroxychalcone (DHCHC)

2,4-Dihydroxy-5-acetylacetophenone (1) was prepared by the reaction of acetic anhydride and resorcinol in presence of anhydrous $ZnCl_2$. The product was obtained as colourless crystalline solid, m.pt. 178-80°C, (yield 80%).

1 Gm of compound (1) was mixed with salicylaldehyde (2 ml) in ethanol (20 ml) and aqueous KOH (10 g in 10 ml of water) was added. The mixture was allowed to stand for 24hrs. On acidification with 1:1 HCl followed by cooling in ice, a solid product was obtained. This was then recrystallised from benzene when yellow needle shaped crystals were obtained.

2',4'-Dihydroxy-5'-(4-Hydroxy-3-Methoxy) Cinnamoyl-4-Hydroxy-3-Methoxychalcone (DHCHMC):

Compound 1 (1 g) was mixed with vanillin (1.5 g) in ethanol (20 ml) and aqueous KOH (10 g in 10 ml of water) was added. By a similar work up of the mixture as described above the crude product on recrystallisation from benzene gave out as yellow needles.

The respective ligand (10m mol) in absolute ethanol (100 ml) was treated with 1M NaOH (2 ml) and the mixture was then added slowly with constant stirring to a solution of $FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$ (5m mol) in water (200 ml) and kept for 1 hr followed by cooling in ice. The precipitated complexes were filtered, washed with aqueous ethanol (1 : 1 v/v) and dried over fused $CaCl_2$ (yellow colour, yield 70%).

The analytical data show that the complexes have the composition $\text{Fe(L)(H}_2\text{O)}_2$ (L = DHCHC or DHCHMC). Low molar conductance values (15.34–22.50 $\Lambda_m \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$) of Fe(II) complexes of DHCHC and DHCHMC in DMF suggested their non-electrolytic nature. Their μ_{eff} values (ca 5.00 B.M.) corresponded to octahedral geometry. The free ligand showed a weak broad band around 3300–3500 cm^{-1} . But the IR spectra of Fe(II) complexes exhibited an intense broad band near 3400 cm^{-1} due to coordinated water molecules. The δ OH-bands at 1395 cm^{-1} (DHCHC) and 1375 cm^{-1} (DHCHMC) were absent in the complexes due to deprotonation of phenolic OH of the ligand. The $\nu\text{C-OH}$ band at 1200 cm^{-1} showed a negative shift due to bonding to metal ion. The $\nu\text{C=O}$ band at 1640 cm^{-1} in the ligands was shifted to lower frequency at 1610 cm^{-1} in the complexes due to coordination. The $\nu\text{M-O}$ band was observed at 600 cm^{-1} (DHCHC) and 570 cm^{-1} (DHCHMC) in the complexes.

ACKNOWLEDGEMENT

The authors are thankful to the Head of the Department for his encouragement. They also thank Prof. M.S. Tempesta, Director, Analytical Service Centre, University of Missouri for PMR data.

REFERENCES

1. N.S. Biradar, B.R. Patil and V.H. Kulkarni, *Curr. Sci. (India)*, **45**, 203 (1976).
2. M. Palaniandavar and C. Natarajan, *Aust. J. Chem.*, **33**, 737 (1980).
3. V.R. Venkataraman and S. Nagarajan, *J. Indian Chem. Soc.*, **63**, 925 (1986).
4. A.S.R. Anjaneyulu, A.V.R. Ramprasad and D.S. Reddi, *Curr. Sci., (India)*, **48**, 300 (1979).

(Received: 7 November 1989; Accepted 14 July 1990)

AJC-196