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NOTES

Studies on Co(II) Thiosemicarbazone Adducts with 2-Pyridine Ethanol and 2,2′-Bipyridyl

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The adducts of thiosemicarbazones of 2,5-dimethoxy phenyl glyoxal, 2-hydroxy-5-carboxy acetophenone and Co(II) phenyl thiosemicarbazone of resacetophenone with 2-pyridine ethanol and 2,2′-bipyridyl with the composition ($CoLL'Cl_2H_2O$), ($CoLL''Cl_2H_2O$), $CoLL'Cl(H_2O)_2$ and $CoLL''Cl(H_2O)_2$, where L=thiosemicarbazone and L′=2-py. ethanol and L″=2,2′-bipy., have been prepared. The adducts have been characterised on the basis of magnetic and spectral studies. Conductance values in DMF showed them to be non-electrolytes.

2-Hydroxy-5-methylacetophenone thiosemicarbazone has been used as indicator in complexometric titrations of Fe(III) with EDTA.[1] The present paper reports the adducts of Co(II) thiosemicarbazones of 2,5-dimethoxy phenyl glyoxal, 2-hydroxy-5-carboxy acetophenone and Co(II) phenyl thiosemicarbazone of resacetophenone with 2-pyridine ethanol and 2,2′-bipyridyl.

2,5-Dimethyl phenyl glyoxal[2], 2-hydroxy-5-carboxy acetophenone[3,4] and resacetophenone were prepared as reported earlier.[5] The thiosemicarbazones or phenyl thiosemicarbazone were obtained as yellow solids by refluxing an ethanolic solution of these compounds with thiosemicarbazide or its phenyl derivative for 3–4 hrs on a water bath.

Equimolar solutions of $CoCl_2 \cdot 6H_2O$, thiosemicarbazone/phenyl thiosemicarbazone (in ethanol) and 2-pyridine ethanol or 2,2′-bipyridyl were mixed in 1 : 1 : 1 ratio and refluxed for 2–2.5 hrs. Light pink solids separated out on cooling, were washed with ethyl alcohol and dry ether and dried.

The adducts are insoluble in common organic solvents but fairly soluble in DMF. Analytical reports are within the $\pm 1\%$ error and showed 1 : 1 : 1 stoichiometric composition. The conductivity of the adducts in DMF (6–11 $ohm^{-1}\ cm^2\ mol^{-1}$) indicated their non-electrolytic nature. Magnetic moments of Co(II) adducts lie in the range 4.99–5.09 B.M.

The weak ν C=S band found in the ligands at 1175 cm^{-1} gets shifted to lower frequency band (1155–1160 cm^{-1}) in the adducts. ν C=N band appearing in the spectra of ligands at 1625 cm^{-1} has been found to be shifted to *ca.* 1590 cm^{-1}. ν C=O in the ligand found at 1650 cm^{-1} gets

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shifted to *ca.* 1630 cm^{-1} in the 2,5-dimethoxy phenyl glyoxal thiosemicarbazone adducts indicating the participation of C=O in coordination. In the rest of the adducts, the band characteristic of phenolic OH found in the thiosemicarbazones except that of 2,5-dimethoxy phenyl glyoxal is missing showing the deprotonation of –OH and involvement of oxygen atom in coordination. A shift of 3650–3584 cm^{-1} band to higher frequency band in adducts is suggestive of bonding of metal ion through oxygen of OH group. A band at 620 cm^{-1} has been assigned to the deformation of pyridine. The bands attributed to 2,2'-bipyridyl are also modified. Thus both these heterocyclic compounds act as bidentate ligands in the adduct formation. Broad band at *ca.* 3450 and a band at 820 cm^{-1} is identified in the spectra of adducts, due to coordinated water. A band in lower frequency region (220–210 cm^{-1}) and bands at *ca.* 500 and 450 cm^{-1} could be due to ν(M–Cl), ν(M–N) and ν(M–O) respectively.

REFERENCES

1. J. R. Shah and R. P. Patel, *J. Indian Chem. Soc.*, **50**, 157 (1973).
2. A. I. Vogel, A Text book of Practical Organic Chemistry, 4th Edition, Longman, London, 440 (1978).
3. ———, A Text Book of Practical Organic Chemistry, 3rd Edition, Longmans, London, 996 (1956).
4. D. N. Shah and N. M. Shah, *J. Indian Chem. Soc.*, **26**, 235 (1949).
5. E. C. Horning, Organic Synthesis, Coll. Vol. 3, p. 761, John Wiley and Sons Inc., N.Y.

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