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COPPER (II) COMPLEXES OF ONO DONOR TRIDENTATE LIGANDS N-(HYDROXYALKYL)-2-HYDROXYNAPHTHYLIDENEIMINES

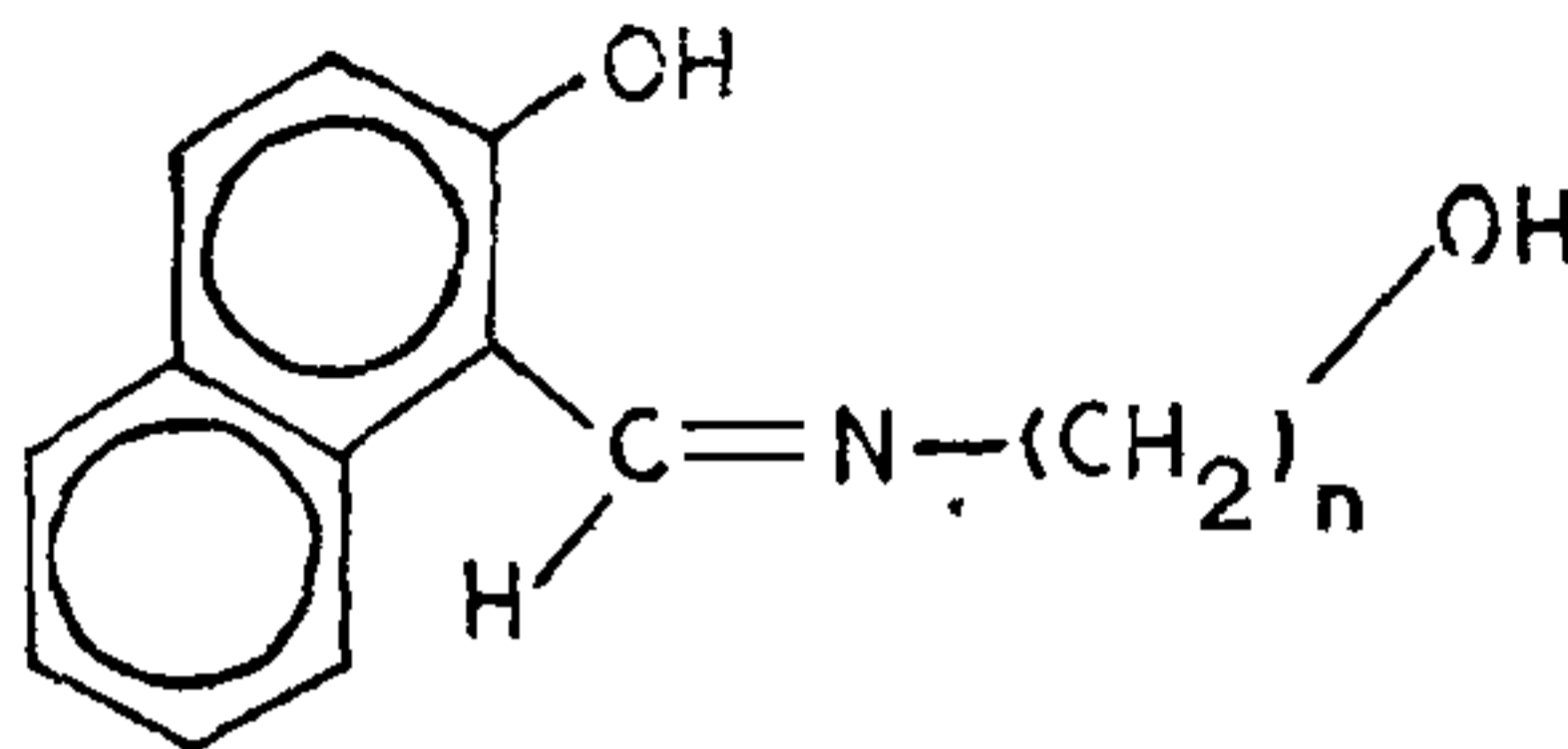
THERE has been considerable interest in recent years on the syntheses and magnetic properties of metal (II and III) complexes with tridentate dibasic ligands¹⁻³. The tridentate dibasic character of these ligands force the metal (II and III) ions to dimerise or polymerise leading to metal complexes with unusual magnetic and structural properties. We report in this communication the synthesis of copper (II) complexes of ONO donor tridentate dibasic Schiff bases (I) derived from 2-hydroxy-1-naphthaldehyde and alcoholamines, viz., ethanolamine, propanolamine and isopropanolamine.

Synthesis of the Complexes

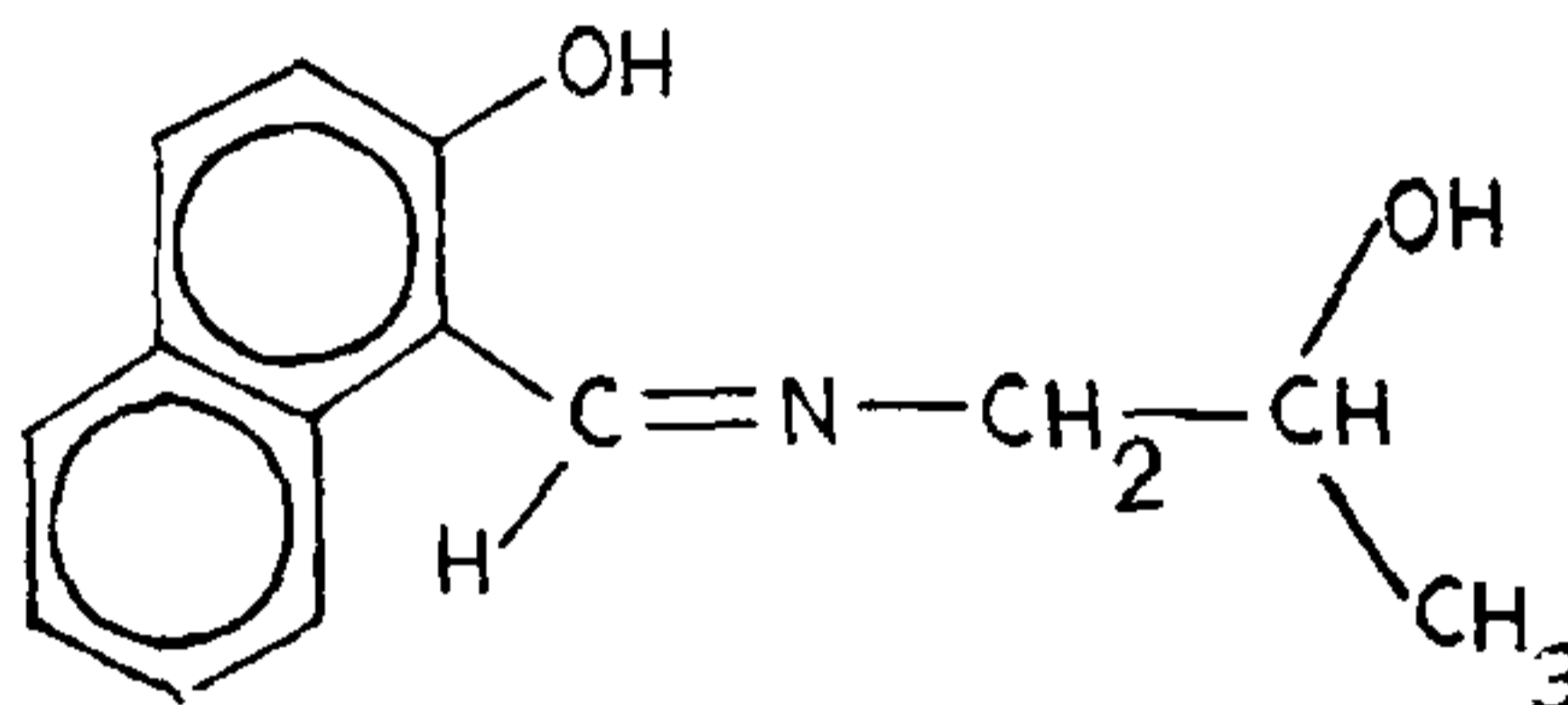
Copper (II) acetate monohydrate (0.005 mole) was dissolved in 60 ml of hot methyl alcohol. To this a methyl alcoholic solution (45 ml) of the appropriate condensed Schiff base (0.005 mole) was added and the mixture was refluxed for 2 hr. The green precipitates formed were filtered under reduced pressure, washed with methyl alcohol and dried under vacuum. The complexes are insoluble in common non-coordinating organic solvents. The analytical and characterisation data of the complexes are presented in Table I.

The data indicate 1:1 metal:ligand ratio in the complexes. The infrared spectra of the complexes

do not exhibit the $\nu(\text{OH})$ stretch and this is indicative of the dibasic behaviour of the ligands in the complexes. The magnetic moments of the complexes, copper (hydroxynaphthaldehyde-ethanolamine) and copper (hydroxynaphthaldehyde-isopropanolamine)



I a. $n = 2, 3$.



determined by the Gouy method, increase as the temperature is lowered from 297 to 83° K. The exchange integral, J of the complexes calculated using the Bleaney-Bowers equation⁴ is + 67 and + 28 cm^{-1} respectively. The positive J values and the increase of the magnetic moment with lowering of temperature are indicative of the presence of ferromagnetic exchange in these complexes^{5,6}. The magnetic moment of the complex copper (hydroxynaphthaldehyde-propanolamine) is 0.46 B.M. at 297° K and the moment shows little temperature dependence. J of this complex is -846 cm^{-1} . The negative J value and low magnetic moment are indicative of the presence of antiferromagnetic exchange in this complex¹. This complex is a novel example where the spins of the interacting copper (II) ions are completely coupled having a sole population of the diamagnetic singlet state. Only few such copper (II) complexes with sole population of the singlet ground state have been reported in the literature⁷. The magnetic properties of Cu (hydroxynaphthaldehyde-propanolamine) and the other two complexes containing ethanolamine and isopropanolamine are different and it is likely that the structures of the antiferromagnetic and ferromagnetic complexes are different¹.

TABLE I
The analytical and characterisation data of copper (II) Schiff base complexes^{a,b}

Complex	%Cu	%N	Temp. (°K)	χ_M^{corr} (10^{-6} cgs unit)	μ_{eff} (B.M.)	J_e cm^{-1}
Cu (hydroxy-ethanolamine) $\text{CuC}_{13}\text{H}_{11}\text{NO}_2$	Found: 22.1	4.9	297	1489	1.89	+67
	Reqd.: 22.96	5.06	155	2998	1.94	
			85	5805	2.01	
Cu (hydroxy-propanol-amine) $\text{CuC}_{14}\text{H}_{13}\text{NO}$	Found: 21.0	5.2	297	88	0.46	-846
	Reqd.: 21.86	4.82				
Cu (hydroxy-isopropanol-amine) $\text{CuC}_{14}\text{H}_{13}\text{NO}_2$	Found: 21.0	4.9	297	1352	1.84	+28
	Reqd.: 21.86	4.82	118	3702	1.88	
			83	6048	2.01	

^a Abbreviation: hydroxy = 2-hydroxy-1-naphthaldehyde.

^b The magnetic moment was calculated using the Curie equation: $\mu_{\text{eff}} = 2.84 (\chi_M^{\text{corr}} \times T)^{1/2}$ B.M.

^c Temperature independent paramagnetism term = 60×10^{-6} cgs. unit, $g = 2.1$.

The synthesis and characterisation of copper (II) complexes using other alcoholamines like 2-amino-1-butanol, 4-amino-1-butanol and 2-amino-2-methylpropanol are in progress. The corresponding oxovanadium (IV) complexes are also being studied in order to compare the magnetic properties of these $s = \frac{1}{2}$ systems.

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CHEMICAL EXAMINATION OF *TECOMELLA UNDULATA* (G. DON) SEEM

Tecomella undulata (G. Don) Seem (Bignoniaceae) is described to be useful^{1,2} in curing urinary discharges, enlargement of spleen, gonorrhoea, leucoderma and liver diseases. Its bark has been the subject of extensive chemical study³⁻⁴. In continuation of our earlier work^{5,6} on extractives from its heartwood, we now report the isolation and identification of veratric acid; which is the first report of its occurrence in free state from the heartwood of a tree.

Air dried and coarsely powdered heartwood (550 g) was successively extracted with petroleum ether 60-80° and acetone. A good yield of lapachol⁵ was obtained from petrol extract. Acetone extract on concentration and keeping in refrigerator gave a solid (tecomelloside)⁶. Latter was removed by decantation. From the mother liquor, solvent was completely removed and the deep orange-red mass obtained was separated into ether-soluble (1.34 g) and ether-insoluble (3.15 g) fractions. The insoluble fraction contained mainly the glucoside. The ether-soluble fraction resisted crystallisation and therefore was subjected to column chromatographic resolution over deactivated silicagel. From ethylacetate-chloroform (2:3 and 1:1) fractions, after removal of solvent an orange-yellow solid (0.49 g) was obtained and was further purified by filtration of its chloroform solution through a column of silicagel and then crystallised from ethyl acetate-petroleum ether 60-80° (1:1) as needles, mp. 181-182° (Found: C, 59.45; H, 5.63; OCl_3 , 34.20; Calcd. for

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