

Neutral Ligand Complexes of Alkali Metal Salts of Some Organic Acids with 2-Methyloxine

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— A number of novel neutral ligand complexes having general formula $ML \cdot HL'$ have been prepared where $M = Li, Na$ or K , $HL' = 2$ -methyloxine. This neutral ligand complexes has been characterised on the basis of elemental analysis, IR spectral studies and conductance measurement.

INTRODUCTION

2-Methyloxine has been extensively investigated for its possible use in analytical determination of metal ions¹⁻³. Its complexes with transition metals as well as rare earth are well investigated⁴⁻⁶. Present study deals with investigation of the coordination capability of these two ligands towards alkali metals.

EXPERIMENTAL

The ligand 2-methyloxine (MeHL) was prepared by reported method. Alkali metal salts of 2-methyloxine were prepared according to the method reported previously.

Preparation of complexes

Suspension of the alkali metal salt (ML) of the ligand HL with the ligand HL was taken in slight excess in absolute ethanol. The solution was refluxed for some time when uniform solution was obtained. A change in the colour of the solution suggest the possibility of complex formation. The contents were slightly concentrated and cooled when the coloured adduct got separate. These were filtered, washed with ethanol or ether, dried in an electric oven at 80°C and stored in a desiccator over anhydrous calcium chloride.

RESULTS AND DISCUSSION

Table-1 lists the physical properties of the ligand (HL), its alkali metal salts (ML) and the new neutral complexes ($ML \cdot nHL$); where $M = Li, Na, K, Rb$ or Cs , $n = 1$ and 2 , $HL = 2$ -methyloxine and $L =$ respective anions of (HL) ligand. The lower values of molar conductance ($6.9-8.1 \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$) of the complexes suggest the non-electrolytic nature.

The spectra of 2-methyloxine shows medium broad absorption bands at 3400 cm^{-1} . The presence of absorption feature in these region points out to the presence of hydrogen bonding.

TABLE-1. ANALYTICAL DATA OF ALKALI METAL COMPLEXES.

Compound/(colour)	m.p./decomp./ transition temp. (°C)	% Found/(Calcd.)			
		C	H	N	M
MeHL(2-methyloxine) (White)	74 m				
LiMeL.MeHL (White)	130 t	73.50 (74.07)	5.50 (5.24)	8.62 (8.64)	2.00 (2.16)
NaMeL.MeHL (White)	140 t	70.80 (70.58)	4.90 (5.00)	8.20 (8.23)	6.90 (6.76)
KMeL.MeHL (Cream)	170 t	66.80 (67.41)	4.90 (4.77)	7.80 (7.86)	10.82 (10.95)
RbMeL.(MeHL) ₂ (Cream)	192 t	64.70 (64.11)	4.90 (4.63)	7.50 (7.47)	16.80 (15.22)
CsMeL.(MeHL) ₂ (Pale yellow)	198 m	61.22 (59.12)	4.60 (4.26)	7.30 (6.89)	22.40 (21.82)

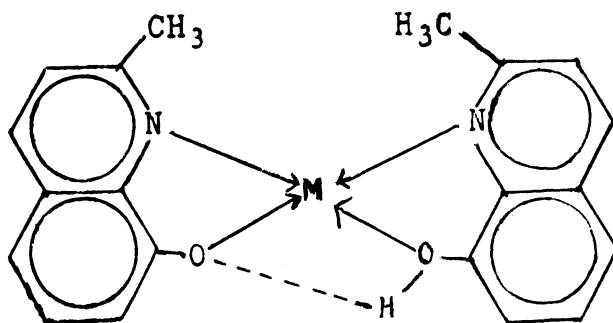
Neither the broad absorption feature nor the $\nu(\text{O—H})$ above 3000 cm^{-1} are observed in the methyloxine complexes. The band at 1605 cm^{-1} occurs due to $\nu(\text{C=N})$ is shifted down by $15\text{--}30\text{ cm}^{-1}$ in the alkali metal complexes. For the rubidium and caesium complexes an additional band at 1555 cm^{-1} is observed. It is possible that the smaller cations (Li, Na) are tetrahedrally coordinated, while the larger ones (Rb, Cs) are octahedrally coordinated. Substitution of a methyl group in the 2-position in 8-hydroxyquinoline would seem not to affect the nature of the complexes formed. Presumably steric hindrance only affects octahedral coordination in the small group III cation *e.g.* Al^{3+} . The metal ion reacts primarily with the negatively charged oxygen to form essentially ionic bonds. However, some degree of interaction may well occur with the covalent nitrogen, especially since no bands characteristic of $\text{N}\dots\text{H}\dots\text{O}$ are observed in the region $2680\text{--}2050\text{ cm}^{-1}$. The absence of N—H bands suggest that the H-bond is between two oxygen atoms, and that the ligands must therefore be in a *cis* configuration. However, since no band has been definitely assigned to the $\text{O}\dots\text{H}\dots\text{O}$ stretching frequency, this might be considered a mute point.

TABLE-2. PERTINENT IR DATA (cm^{-1}) FOR LIGAND AND THEIR NEUTRAL COMPLEXES WITH ALKALI METALS

Compound	—OH	> C=N
MeHL	3400 br	1605 m
LiMeL.MeHL	—	1580 m
NaMeL.MeHL	—	1590 m
KMeL.MeHL	—	1585 w
RbMeL.(MeHL) ₂	—	1570 s, 1555 m
CsMeL.(MeHL) ₂	—	1575 m, 1555 m

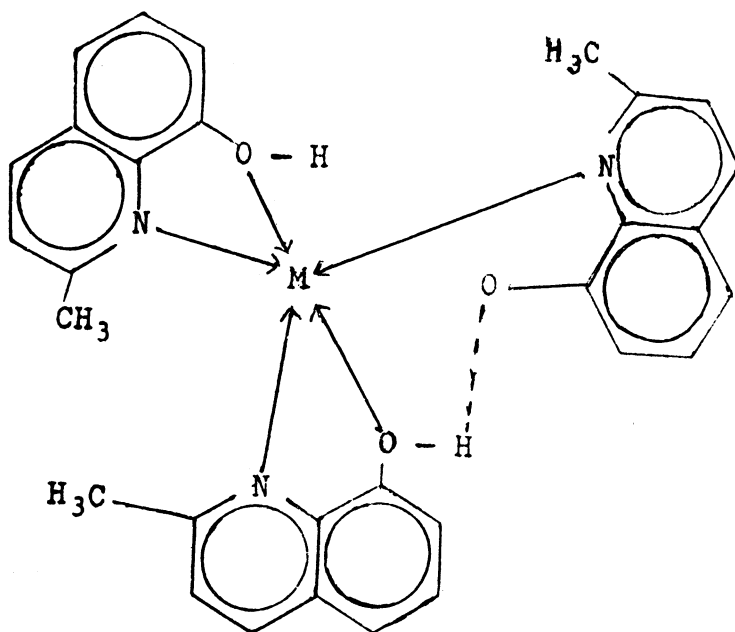
m = medium, s = strong, b = broad, w = weak

On the basis of foregoing studies, the probable structures of the complexes are given in Fig. I & II.



where $M = \text{Li, Na or K}$

Fig. I



where $M = \text{Rb or Cs}$

Fig. II

REFERENCES

1. M. De Clerq and C. Duval, *Anal. Chim. Acta*, **5**, 290 (1951).
2. J.P. Phillips and F.J. O'Hara, *Anal. Chim. Acta*, **23**, 535 (1951).
3. M. Borrel and R. Paris, *Anal. Chim. Acta*, **6**, 395 (1952).
4. J.P. Phillips and L.L. Merritt, *J. Am. Chem. Soc.*, **6**, 3984 (1949).
5. J.P. Phillips J.F. Emery and H.P. Price, *Analyt. Chem.*, **24**, 1033 (1952).
6. M. Borrel and R. Paris, *Analyt. Chem.*, **5**, 573 (1951).

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