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Mixed ligand complexes of rubidium & caesium metal salts

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Abstract

The paper presents the synthesised and characterised complexes of Rb and Cs metal salts of some organic acids viz o-nitrophenol, 2,4-dinitrophenol, 1-nitroso-2-naphthol & 8-hydroxy quinoline with o-aminothiophenol having as co-ordination sites N and S atoms.

Introduction

Mixed ligand complexes of type ML₂HL', have been synthesised and characterised where M = Rb or Cs; L = deprotonated 1-nitroso-2-naphthol (IN₂N), 8-hydroxy quinoline (8HQ), o-nitrophenol (ONP), 2, 4-dinitrophenol (DNP) and HL' = thiosalicylic acid have been synthesised and characterised on the basis of elemental analysis, conductivity measurement and infrared spectral studies. IR spectra suggested that hydrogen bonding is not necessary for formation of these complexes.

Complexing abilities of salicylic acid have been investigated [1-3]. Literature survey reveals that a number of mixed ligand complexes of alkali and alkaline earth metals with the ligand have been reported [4-7].

Material and Methods

1-Nitroso-2-naphthol, 8-hydroxyquinoline, o-nitrophenol, 2, 4-dinitrophenol of AnalaR grade were taken. O-aminothiophenol of E.M. grade was distilled at 23°C and when cooled, its needle like crystals (m.p. 26°C) were obtained.

Preparation of Rb and Cs metal salts of organic acids: Equimolar proportion of RbOH or CsOH and organic acid were refluxed in absolute ethanol medium for about 30 minutes on a water bath. The clear solution cooled to get the characteristic colour precipitate of Rb or Cs metal salt. It was filtered, washed with solvent and dried in an electric oven at 80°C.

Preparation of the Complexes: 1:1 stoichiometric amount of rubidium or caesium metal salt of organic acid was refluxed in absolute ethanol for 1-2 hour with constant stirring, then cooled which led the coloured complex precipitated out. The precipitated complex was filtered, washed with solvent and dried in electric oven at 80°C.

Results & discussion

Some physical properties of the ligand and its mixed ligand complexes are listed in Table-1.

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Table 1: Ligand and its mixed ligand complexes Analysis

Compound	Colour	M.p. Decomp. Trans. Temp. (°C)	Conductivity	Analysis % (Found/Calcd.)			
				C	H	N	M
ThsalA	Pale cream	163 m	5.0	–	–	–	–
Rb (1N2N). ThsalA	Reddish brown	178 d	16.0	49.65	2.99	3.49	20.73
				(49.63)	(2.92)	(3.40)	(20.68)
Cs (1N2N). ThsalA	Reddish brown	170 d	20.0	44.49	2.69	3.11	29.04
				(44.44)	(2.61)	(3.05)	(28.95)
Rb (8HQ). ThsalA	Cream	193 d	15.0	50.17	3.20	3.73	22.23
				(50.13)	(3.13)	(3.65)	(22.12)
Cs (8HQ). ThsalA	Cream	185 d	18.0	44.64	2.83	3.31	31.12
				(44.54)	(2.76)	(3.25)	(30.84)
Rb (ONP). ThsalA	Yellow	200 d	17.0	41.54	2.70	3.80	22.89
				(41.36)	(2.65)	(3.71)	(22.55)
Cs (ONP). ThsalA	Yellow	190 d	20.0	36.85	2.41	3.47	31.40
				(36.70)	(2.35)	(3.27)	(31.27)
Rb (DNP). ThsalA	Deep Yellow	195 d	15.0	37.06	2.16	6.66	20.24
				(36.94)	(2.13)	(6.63)	(20.14)
Cs (DNP). ThsalA	Deep Yellow	183 d	18.0	33.25	2.00	5.63	26.35
				(33.19)	(1.91)	(5.55)	(26.27)

*Molar conductivity $\text{ohm}^{-1} \text{mol}^{-1} \text{cm}^2$ of 10^{-3}M solution in MeOH.

Almost all complexes have been found to be coloured and stable in dry air but decomposed in moist air. They are soluble in polar solvents e.g. MeOH, EtOH, DMF but insoluble in non-polar solvents. From result, it was evident that all these complexes either decompose or undergo transformation at temperature which are considerably higher than the melting point of the ligand, indicating their greater thermal stability.

Conductivities: Molar conductivities of the ligand (Thiosalicylic acid) and its mixed ligand complexes of rubidium and caesium metal salts of different organic acids measured on Sys-tronics Digital Direct Reading Conductivity Meter-304 at 30°C in 10^{-3}M MeOH solution.

The conductivity values are given in Table-1. Significantly low values of molar conductance (15.0 to 20.0) of mixed ligand complexes of Rb and Cs suggest them to be non-electrolyte (i.e. covalent).

Infrared spectra: Infrared measurements for the ligand (ThsalA) and its hitherto unknown mixed ligand rubidium and caesium complexes of the type $\text{ML.HL}'$, where M = Rb or Cs, L = deprotonated ONP, 8HQ, ONP, DNP and HL' = thiosalicylic acid were recorded in the region between $4000\text{--}650 \text{cm}^{-1}$ in KBr phase with the help of spectrophotometer. Pertinent IR data for these compounds were recorded in Table-2.

Table 2: Compound & Selected IR absorption bands

Compound	Selected IR absorption bands (in cm^{-1})	
	$\nu\text{S-H}$	νCOOH
ThsalA	2530 m	1690 s
Rb (1N2N). ThsalA	2350 m	1610 sh, 1590 s, 1580 m, 1550 m
Cs (1N2N). ThsalA	2480 m	1600 sh, 1580 s, 1570 m
Rb (8HQ). ThsalA	2380 m	1600 sh, 1560 m, 1500 m
Cs (8HQ). ThsalA	2500 w	1590 s, 1580 m, 1530 m
Rb (ONP). ThsalA	2381 w	1590 s, 1580 m, 1550 m
Cs (ONP). ThsalA	2371 m	1600 s, 1580 m, 1540 m
Rb (DNP). ThsalA	2351 m	1600 sh, 1590 s, 1550 m
Cs (DNP). ThsalA	2381 m	1670 s, 1590 m, 1560 w

s = strong, w = weak, m = medium, sh = shoulder

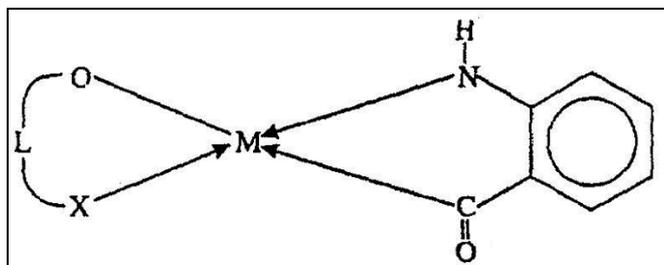
The spectra of the mixed ligand complexes differ from that of acid metal salts and the second ligand (Thiosalicylic acid). While the spectrum of the ligand contains a moderately medium: absorption band at 2530cm^{-1} , the region of S-H vibration frequency, this band has shifted down by $30\text{--}70 \text{cm}^{-1}$ in the complexes, indicating thereby that coordination has taken place; through S-atom of S-H group.

The strong band at 1680cm^{-1} in thiosalicylic acid assigned to -COOH stretching frequency in unionized -COOH is splitted in two or more medium intensity bands in the complexes. One of the bands appears at 1600cm^{-1} , which may be due to the chelation of carboxylic acid group. The remaining other bands in the region $1500\text{--}1550 \text{cm}^{-1}$ occur

due to the presence of -NO , -NO_2 or -CN groups of rubidium and caesium metal salt of first ligand such as 1-nitroso-2-naphthol (1N2N), 8-hydroxyquinoline (8HQ), o-nitrophenol (ONP), 2, 4-dinitrophenol (DNP). The shifting and splitting of the -COOH band suggests that coordination has taken place through oxygen atom of carboxylic acid (-COOH) group.

Structure and bonding

The probable structure on the basis of above studies, can-produced and schematically given as below:



(where M = Rb or Cs, X = O or N)

Fig 1: probable Structure and bonding

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