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Studies of mixed ligand complexes of rubidium and caesium metal salts of some organic acids

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Abstract

The present study was aimed at 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 a coordination sites N and S atoms.

Keywords: caesium metal, rubidium, ligand

Introduction

Complexes of transition metals[1–3] as well as non–transition metals[4–5] with systems containing both nitrogen and sulphur donors have been reported. In this paper, Complexes of the type ML.HL', have been synthesised and characterised, where M=Rb or Cs, HL' = o-aminothiophenol, L=deprotonated o–nitrophenol (ONP), 2, 4– dinitrophenol (DNP), 1–nitroso–2–naphthol (1N2N) & 8–hydroxy quinolin (8HQ). All the complexes have been found to be four coordinated in which ligand o–aminothiophenol is coordinated through N–atom of amino (NH_2) group & S–atom of the thiol (–SH) group forming five membered stable chelation.

Material and Methods

1–Nitroso–2–naphthol (1N2N), o–nitrophenol (ONP), 2, 4–dinitrophenol (DNP) and 8–hydroxyquinoline (8HQ) of Anal R grade were taken, O–aminothiophenol of E.M. grade was distilled at 23^0C and when cooled, its needle like crystals (m.p. 26^0C) were obtained Equimolar proportion of RbOH or CSOH and organic acid were refluxed in absolute ethanol medium in a conical flask 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 a electric oven at 80^0C. Stoichiometric amount of rubidium or caesium metal salt of organic acid & thiosalicylic 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 80^0C.

Results and Discussion

Conductivity of the ligand, o–aminothiophenol and its mixed ligand complexes of rubidium & Caesium metal salts of different organic acid were measured by Systronics Digital Reading Conductivity Meter–304 at 30^0C in 10^{-3} M DMF solution. The conductivity values are given Table–1

Table 1

Compound	Colour	M.p. Decomp. Trans. Temp. (^{0}C)	Conductivity	Analysis % (Found/Calcd.)			
				C	H	N	M
O–aminothiophenol (OATP)	Colourless	24 m	5.0	–	–	–	–
Rb (1N2N) (OATP)	Deep brown	205 d	16.0	50.0 (50.26)	3.44 (3.35)	7.56 (7.52)	21.30 (22.25)
Cs (1N2N) (OATP)	Deep brown	193 t	20.0	44.65 (44.63)	3.05 (3.07)	6.61 (6.51)	22.30 (22.23)
Rb (8HQ) (OATP)	Light brown	205 t	1.0	50.91 (50.83)	3.70 (3.67)	7.95 (7.70)	24.06 (24.01)
Cs (8HQ) (OATP)	Light brown	200 d	20.0	44.81 (44.75)	2.30 (3.23)	6.99 (6.96)	32.13 (30.06)
Rb (0NP) (OATP)	Brownish Yellow	196 t	14.0	41.42 (41.36)	3.20 (3.16)	6.09 (6.04)	24.51 (24.42)
Cs (0NP) (OATP)	Brownish Yellow	190 t	15.0	36.40 (35.33)	2.80 (2.77)	7.17 (7.04)	33.61 (33.58)
Rb (DNP) (OATP)	Brownish Yellow	208 d	10.0	36.67 (36.64)	2.58 (2.54)	10.71 (10.68)	21.66 (21.61)
Gs (DNP) (OATP)	Brownish Yellow	200 d	14.0	32.70 (32.63)	2.30 (2.24)	9.55 (9.50)	30.21 (30.14)

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

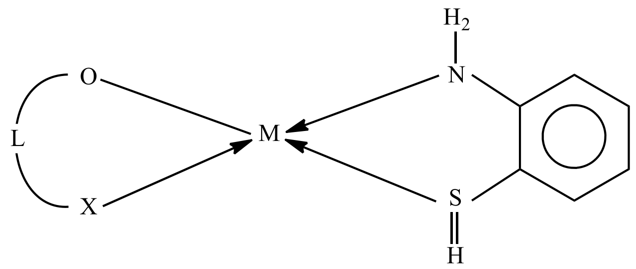
Almost all complexes have been found to be coloured & stable in dry air but decomposed in moist air. They are soluble in polar solvents e.g. MeOH, EtOH, DMF but insoluble in non–polar solvents. From result, it was evident that all these complexes either melting point of the ligand, indicating their greater thermal stability. Infrared spectral of the ligand (o–aminothiophenol) and its hitherto un–known mixed ligand rubidium and caesium metal complexes of general formula, ML.HL', where M=Rb or Cs, L = deprotonated o–nitrophenol, 2, 4–dinitrophenol, 1–nitroso–2–naphthol, 8–hydroxyquionoline & HL' = o–aminothiophenol have been recorded in region 4000–650 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 (in cm^{-1})		
	^{V}–N–H	^{V}S–H	^{V}–NH$_2$
O–aminothiophenol (OATP)	3200 s, 3100 m	2350 m	1490 s
Rb (0NP) OATP	3170 m, 3075 m	2300 m	1400 s, 1360 m
Cs (0NP) OATP	3170 m, 3075 m	2320 m	1400 s, 1370 m
Rb (DNP) OATP	3180 s, 3090 m	2310 m	1400 s, 1360 m
Cs (dNP) OATP	3170 m, 3070 m	2300 m	1400 s, 1360 m
Rb (1N2N) OATP	3150 m, 3070 m	2300 m	1400 s, 1375 m
Cs (1N2N) OATP	3170 m, 3075 m	2290 m	1400 s, 1380 m
Rb (8HQ) OATP	3180 m, 3027 m	2310 m	1400 s, 1370 m
Cs (8HQ) OATP	3150 m, 3080 m	2100 m	1400 s, 1380 m

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

The absorption range of N–H has been observed in the mixed ligand complexes of rubidium and caesium metals, which might be due to coordination of –NH$_2$ group to the rubidium & caesium metals. As shown in fig.



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

Conclusion

The spectrum of the ligand contains a moderately medium absorption band at 2550 cm^{-1} the region of S–H vibration frequency, this band has shifted down by ~ 40–70 cm^{-1} in the mixed ligand rubidium & caesium metal complexes, indicating there by the coordination has taken place through S–atom of S–H group.

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