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Analysis of meta and para-tolyl substituted tetrazoline-5-thione pd(ii) complexes in acidic

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Abstract

The main objective in this paper is to the some pd(II) complexes of meta & para-tolyl substituted tetrazoline-5-thione have prepared at different Ph(1-2, 5-6, 9-10 & 12-13) & characterised by using different physic-chemical data.

Keywords: meta and para-tolyl substituted, tetrazoline-5-thione pd(ii), in acidic

Introduction

There are many studies in this area. The complexes of pd(II) with meta-&Para-tolyl substituted tetrazoline-5-thione ^[1-2] in acidic & basic medium are very important. In this paper, comparative studies of these complexes are described on the basis of I.R. and ¹H NMR spectra.

Material and Method

The palladium chloride (0-1g) was dissolved in hot dil. HCl and was filtered. 0.2g of 1-meta- & 1 para-tolyl substituted tetrazoline-5-thione were dissolved separately in about 10ml of ethanol & filtered. Two filtrates were mixed & the pH of the two different solutions were made between (9-10) and (12-13).

Similarly, the complexes of 1-meta-and para-tolyl substituted tetrazoline-5-thione were made between off (1-2) & (5-6).

Result and Discussion

The complexes of 1-meta-&1-para tolyl tetrazoline-5-thione in acidic & basic medium show that in acidic medium chloro group is not co-ordinate with pd(II) where was in basic medium the chloro group is co-ordinate with pd(II).

Following pd(II) complexes³ at different pH are given in table-1.

Table 1: pd(II) complexes at different pH

S. No.	Complex	pH
1.	[pd(1-m-TT5TH) ₂ (OH) ₂]C ₂ H ₅ OH	(1-2)
2.	[pd(1-p-TT5TH) ₂ (OH) ₂]	(5-6)
3.	[pd(1-p-TT5TH) ₂ (OH)Cl] ₂ H ₂ O	(9-10)
4.	[pd(1-m-TT5TH)(H ₂ O)CL ₂] ₂ H ₂ O	(12-13)

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Table 2: Comparative studies of IR & ¹H NMR Spectra of Complex

Complex (Color, m. p.)	[pd(1-mTT5TH) ₂ (OH) ₂] C ₂ H ₅ OH (orange, 215 °C)	[pd(1-p-TT5TH) ₂ (OH) ₂] (light orange, 170 °C)	[pd(1-p-TT5TH) ₂ (OH)Cl]2H ₂ O (light brown, 201 °C)	[pd(1-m-TT5TH)(H ₂ O)Cl ₂]8H ₂ O (Brown, 201 °C)
Analysis (%) (pd)	18.8(18.5)	20.6(20.2)	18.6(18.3)	25.0(25.0)
Found (calcd.) (c)	36.8(37.8)	37.3(36.6)	32.9(33.1)	22.2(22.6)
(H)	4.4(4.2)	3.2(34)	3.4(3.6)	3.2(3.3)
I.R. Bands (cm₋₁)				
V _{OH}	3440 mbr, 3217 mbr	2900w	2950vw	–
vH ₂ O (Co-ordinated)	–	–	–	3411mbr
V _{NH} + δ _{CH} (aromatic)	2940vw, 2765s	2950vw	3070vw(b)	2950vw, 2775s
vH ₂ O (Lattice water)	–	–	3270w, 3190w	3270w, 3180w
V _{CS} + v _{CN} (thioamide band I)	1640vw	1525s	1530s	1650w
δCH ₃	1470vw, 1410m	1410m&b, 1395vw	1405w, 1340vw, 1330vw	1480vw, 1420m
In plane bending mode	1320vw, 1305vw, 1250wb	1330w, 1260w	–	1330vw, 1315vw, 1260wb
5-membered heterocyclic ring vibration	–	2910w, 2080wb	2905wb, 2090wb	–
V _{CN} + V _{NN} + V _{CS} (Thioamide band III)	1170vw, 113vw, 1090m, 1035w, 1000w	1190w, 1095w, 1065w, 1030w, 1010w	1090vw, 1065vw, 1030vw, 1010vw	1180vw, 1140vw, 1100w, 1045w, 101-vw
V _{CS-Pd} (Thioamide band IV)	800vw	760w	760vw	810vw
Out of plane CH vibration	760s, 710m	825s	940vw	770s, 720w
δ _{NCS}	615vw, 575vw, 545vw, 390vw	560vw, 505vw	560vw	555vw, 400vw
V _{pd-OH}	380w	530	545w, 525w	–
V _{pd-Cl}	–	–	350vw	360vw
V _{pd-S}	245w	260w	255w	–
structure	Trans-square planar	Trans-square planar	Trans-square planar	Trans-square planar
¹ Hnmr(ppm)				
8-Phenyl protons	7.5	7.4	7.4	–
4-Phenyl Protons	–	–	–	7.4
6-Protons of two methyl grs.	3.8	2.8	2.9	–
3-Protons of one methyl gr.	–	-2.15	–	3.7
2-Protons of two -OH grs.	2.15	–	–	–
1-Protons of one-OH grs.	–	1.4 & 2.4	1.9	–
2-Imino Protons	1.25	–	1.5 & 2.5	–
1-Imino proton	–	–	–	–
2-Protons of co-ordinated H ₂ O	–	3.6	–	1.3
1-Protons of aromatic-SHgr	–	–	3.5	–

The spectral data [4-7] of these complexes are given in table-2

Infrared spectra: There is V_{CS} + V_{CN} (thioamide bond I) [8-11] spectra found in P-tolyl complexes which are strong ranges from 1525-1530 cm⁻¹ while in m-tolyl complexes, it is very weak ranges from 1640-1650 cm⁻¹. 5-membered heterocyclic ring vibration are found in p-tolyl complexes ranges from (2080 wb-2090wb) cm⁻¹ & (2905w-2910w) cm⁻¹ but such vibration are not found in m-tolyl complexes.

The thioamide band III of the ligand having contribution from V_{CN} + V_{NN} + V_{CS} got split on co-ordination to the Pd(II) ion and different range of bands are observed. This has occurred due to decrease in symmetry of the ligand on co-ordination through 'S'.

There is a very weak band formed in the spectrum of both p-tolyl and m-tolyl complexes at 760 cm⁻¹ and (800-810) cm⁻¹ respectively. This indicates that co-ordination of the ligand to the metal ions has occurred through 'S'-atom.

¹H NMR Spectra: Metal-ligand bonding are further substantiate by proton magnetic resonance spectra of complexes and recorded in CdCl₂/TMS. Table-2 indicates that some complexes have multiplet bands centred at δ = 7.5 and 7.4 ppm which may be assigned to 8-phenyl protons [12] of two co-ordinate ligands which other have multiplet band centered at δ = 7.4 ppm of 4-phenyl protons of one co-ordinated ligand.

Conclusion

The one or two imino protons in table-2 of ligands are probably in two different magnetic environments as they are

observed at δ=1.9, 1.25, 1.4, 2.4, 1.5 & 2.5 ppm. It appears that although the structure of the complex is square planar of two substantiate & phenyl tetrazolines-5 thione ligands are present in the complexes are trans to each other and co-ordination of the ligand has occurred through thiocarbonyl 'S' atom.

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