Synthesis, Characterization and Biochemical Studies of some mixed ligand Rh(I) Complexes with 4-Amino-3-ethyl-5- mercapto-S-triazine

Abstract

Some air stable and non electrolytic complexes of Rh(I) ions with 4-amino-3-ethyl-5- mercapto-S-triazine (AEMTH) have been prepared and characterized by various physic-chemical methods, such as micro analytical analysis, magnetic measurement, conductivity measurement, infrared and electronic spectroscopic studies.Oxidation state of metals in these complexes is determined iodomertrically.On the basis of normal coordinate analysis, the square planar configuration has been assigned for all Rh(I) complexes.The ligand and its complex are screened for their antifungal activities against C. albicanes and these may be classified as mixed antifungal agent.

Keywords: Triazine, Rhodium(I), Physico-chemical analysis, Antifungal

Introduction

Mixed ligand phosphine complexes of Rh(I) are versatile homogenous catalyst for hydrogenation and subject to review[1].Many Rh(I) complexes undergo a variety of reactions and most of which involve either replacement of phosphine ligand or oxidative addition to form Rh(III).[RhHCO(PΦ_3)$_2$] and [RhCl(PΦ_3)$_3$] are very effective as catalyst for highly selective hydrogenation of complicated organic molecules[2-3], which are of great importance in pharmaceutical industry. The present study describes preparation characterization and structural investigation of some new mixed ligand complexes of Rh(I) using [RhCl(PΦ_3)$_3$] as starting material.

Experimental

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Chemicals used were of A.R.grade. [RhCl(PΦ_3)$_3$] was prepared by the method described in literature[4].4-Amino-3-ethyl-5- mercapto-S-triazine was prepared by the method described in literature[5].

All the complexes were prepared by using a general methods. [RhCl(PΦ_3)$_3$] and triazine derivatives were dissolved in 20 ml benzene in appropriate moral ratio.The mixture was stirred on magnetic stirrer for about 2-3 hours at 50^0C and cooled in refrigerator.The complexes were separated out.It was filtered, washed successively with ice-cold alcohol and ether and dried under vacuum.10ml pyridine was added into filtrate and mixture was again stirred for about 1 hr and cooled to separate out pyridyl derivative of Rh(I) complex.

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Corbon, hydrogen and nitrogen analysis were performed by conventional method and result were reported in table1.

Results and Discussion

The complex [RhCl(PΦ_3)$_3$] undergoes displacement reaction in benzene solution. Double displacement reaction took place in drastic condition and took more time.The moleculer weight of complex, as determined cryoscopically, suggests them to be monomeric. Magnetic moment of complexes shows the diamagnetic nature of all complexes indicating d^8 configuration of Rh(I). When suspensions of complexes were treated with iodine solution in CCl_4, the violet colour of iodine was discharged suggesting the presence of Rh(I).However, determination of oxidation state of rhodium in the complexes was found by titration with ceric ammonium sulphate using ferroin as indicator[6].All complexes were treated for two electorn charge.

The diagmgnetic nature of complexes suggests the metal ion is in the singlest ground state [$^1A_{1g}$].The reflectance spectra of all

complexes[7] show three absorption bands around 13790, 18200 and 23650 cm^{-1}.The band at 13790 cm^{-1}is broad weak while those at 18200 and 23650 cm^{-1} are medium intence bands and tentatively assigned as($^1A_{1g}$ -$^3B_{1g}$ or$^1A_{1g}$ -$^3B_{1g}$) for band obtained at 18200 cm^{-1}.The band at 23650 cm^{-1} could not be assigned because the ligand also absorbs in this region.

I.R. spectra of ligands snd complexes are given in table 1.The bands obtained at 3260, 3210 and 3110 cm^{-1} in the I.R. spectrum of ligand are assigned as vNH mode of vibrations[8-9]. These bands are either identical or blue shifted on complexation with Rh(I) ion indicates the uncordination of nitrogen in all present complexes suggest the coordination of Rh(I) ion through sulphur atom of ligand[10]. The presence of non ligand new bands at 1635, 1390, 970, 915, 720, 540, cm^{-1} in I.R. spectra of all Rh(I) complexsx indicates the involvement of PΦ_3 molecules in complesation .The blue shifting of thioamide band [TAB] I, II and III, and sharp red shifting (‑ 30 to 40 cm-1) of band IV suggests the bonding through thio carbonyl sulphur[12-13].It is further supported

by appearance of new band as vRh- S at 300 cm^{-1}vRh- Cl and vRh-P are obtained at 350 and 375 cm^{-}1 respectively[14-15].In plane ring deformation and out of ring deformation bands of pyridine[11] is observed at 604 and 405 cm^{-1} in [Rh(PΦ_3)(Py)(AEMTH)Cl].

On the basis of normal coordinate analysis of complexes all Rh(I) complexes are tentatively configurated as square planar.

Antifungal activities

Ligand and Rh(I) complexes were screened for 96 hrs against C.albicanes using cup plate method[16] at 1000, 100 & 10 ppm concentration. These results are compared wirh carbendazim, a typical commercial fungides.Antifungal activities wrere given in table 1. [Rh(PΦ_3)(Py)(AEMTH)Cl] showed 77% activities at 1000 ppm.Since all Rh(I) complexes showed significant antifungal activities and contain more than one functional group.So, all reported Rh(I) complexes may be classified as mixed antifungal agent.

S.NO.	Compound colour/mp(^{0}C)	Elemental Analysis (cal/exp.)				I.R. Band Position			Antifungal Data (%)		
		C	N	H	Rh	vNH	vSH	Tab IV	10	100	1000
1.	(AEMTH) White(144^{0}C)	41.34 (40.87)	24.11 (24.0)	6.89 (5.96)	------	3260m 3210m 3110s	2450wb	780 m	+	+	+
2.	[Rh(AEMTH)(PΦ_3)$_2$Cl] Light yellow(190^{0}C)	47.69 (46.01)	5.56 (4.92)	3.77 (3.58)	10.22 (9.78)		--------	750m	+	++	+
3.	[Rh(AEMTH)$_2$(PΦ_3)Cl] Brown(178^{0}C)	45.30 (44.83)	16.26 (15.8)	4.50 (4.15)	14.94 (14.19)	3260ms 3210m 3110s	--------	745m	+	+	++
4.	[Rh(AEMTH)(PΦ_3)(Py) Cl] Light Brown (160^{0}C)	51.96 (51.23)	11.22 (11.0)	4.49 (4.03)	16.50 (15.16)	3260m 3210m 3110s	--------	745m	+	++	+++

% Transmission: (-)1-25;(+)26-50;(++)51-75;(+++)76-100;(++++)>100

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