



SPECTRAL AND BIOLOGICAL EVALUATION OF SOME ORGANORUTHENIUM COMPLEXES LIGATED WITH SECONDARY THIOMIDE

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ABSTRACT

Convenient synthesis of a series of six octahedral ruthenium (II & III) complexes having general formula $[\text{Ru}(\text{E}\phi_3)_2\text{L}_2]$, $[\text{Ru}(\text{CO})(\text{E}\phi_3)_2\text{L}]$ and $[\text{RuX}(\text{E}\phi_3)_2\text{L}_2]$ (where, HL = 1-paraethoxy phenyl tetrazoline-5-thione, E = P/As; X = Cl/Br) has been described. The heterocyclic thioamide ligand behaves as monoanionic N,S – chelating bidentate ligand. The structure of complexes have been deduced by elemental analysis, conductivity, magnetic and spectral (IR, electronic and ^1H NMR) analysis. The in vitro antimicrobial activity of the investigated compounds was tested against the bacteria such as *S. aureus*, *B. subtilis* and *E. coli* and fungi *A. Flacus*, *A. Parasiticus* and *C. Albicans*. The data reveal that all complexes are more active than free ligand molecule.

Keywords : Heterocyclic thioamide, ruthenium chelates, bio-activities

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INTRODUCTION

Thioamides are bacteriocidal and widely used for many years in the treatment of mycobacterium tuberculosis, *M. leprae* and *M. avium* complex infections.¹⁻³ They have interesting insights into structure, bonding and versatile coordinating ability.⁴ Henderson and co-workers⁵⁻⁷ have reported the synthesis and structural characterization of various transition metal complexes containing chelating thioamide derivatives. Mohr et al⁸⁻¹⁰ have examined reactions, structures and anti-tumour activity of various complexes containing thioamide ligands. Prior to this work, we have reported synthesis, characterization and bioactivities of complexes of rhodium (I), Iridium (I) and ruthenium (III) with heterocyclic thioamide ligands.¹¹⁻¹² The present study describes synthesis, spectral characterization and biological evaluation of some ruthenium chelates with secondary thioamide ligand. The in vitro antibacterial screening of thioamides ligand and complexes against *S. Aureus*, *B. Subtilis* and *E. Coli* and antifungal activity against *A. Flacus*, *A. Parasiticus* and *C. Albicans* are reported herein.

EXPERIMENTAL

All the chemicals used were AR grade or CP grade. Solvents were dried before use. The ligand, 1-paraethoxy phenyl tetrazoline-5-thione (LH)¹³, precursor complexes, $[\text{RuCl}_2(\text{P}\phi_3)_4]$ ¹⁴, $[\text{RuHCl}(\text{CO})(\text{P}\phi_3)_3]$ ¹⁵, $[\text{RuHCl}(\text{CO})(\text{As}\phi_3)_3]$ ¹⁶, $[\text{RuCl}_3(\text{P}\phi_3)_3]$ ¹⁷, $[\text{RuCl}_3(\text{As}\phi_3)_3]$ ¹⁸ and $[\text{RuBr}_3(\text{P}\phi_3)_3]$ ¹⁹ were prepared by the methods reported literature. All ruthenium (II)²⁰ and ruthenium (III)²¹ complexes were prepared by the ligand substitution reactions following our previous method.

The analysis of carbon, hydrogen and nitrogen were performed at CDRI, Lucknow, India. The IR spectra of ligand and complexes were recorded on a Perkin-Elmer Model 577 spectrophotometer in the range of 4000-200 cm^{-1} as KBr pellets. The magnetic measurements were made on a Gouy balance and the diamagnetic corrections for ligand were applied. The UV and visible spectra of the ligand and complexes were recorded on a Beckmann and Carl Zeiss (Jena) Spectrophotometer. The molar conductance of complexes (10^{-3} M) were measured in DMF using Wiss-Werkstatter Weitehm obb type LBR conductivity meter. ^1H NMR spectra of ligand and complexes were recorded with 90 MHz NMR spectrophotometer using TMS as internal indicator. The complexes were dissolved in CDCl_3 for

recording their ^1H NMR spectra in the range of 0-10 PPM. The number of protons were obtained with the help of internal calibrator.

RESULTS AND DISCUSSION

The analytical data of complexes correspond to the composition given in table 1. The thioamide ligand acts as mononegative bidentate and follow the general formula $[\text{Ru}(\text{P}\phi_3)_2\text{L}_2]$, $[\text{RuCl}(\text{CO})(\text{E}\phi_3)_2\text{L}]$ and $[\text{RuX}(\text{E}\phi_3)_2\text{L}_2]$ where $\text{X} = \text{Cl/Br}$; $\text{E} = \text{P/As}$. These air and moisture stable compounds are soluble in DMF, DMSO, acetonitrile but insoluble in H_2O and Et_2O and molar conductance value in the range of 10.20 – 15.60 $\text{cm}^2\text{mol}^{-1}$ inferring that all are non-electrolyte.²²

Magnetic Moment and Electronic Spectra

All ruthenium (II) complexes are diamagnetic indicating the ground state $^1\text{A}_{1g}$ arising from $\text{T}_{2g}^6\text{e}_g^0$ configuration in an octahedral environment.²³ The excited state corresponding to the $\text{T}_{2g}^5\text{e}_g^1$ configuration are $^3\text{T}_{1g}$, $^3\text{T}_{2g}$, $^1\text{T}_{1g}$ and $^1\text{T}_{2g}$. Hence, four bands corresponding to the transitions, $^1\text{A}_{1g} \rightarrow ^3\text{T}_{1g}$, $^1\text{A}_{1g} \rightarrow ^3\text{T}_{2g}$, $^1\text{A}_{1g} \rightarrow ^1\text{T}_{1g}$ and $^1\text{A}_{1g} \rightarrow ^1\text{T}_{2g}$ are possible in order of increasing energy. The electronic spectra of $[\text{RuCl}(\text{CO})(\text{E}\phi_3)_2\text{L}]$ ($\text{E} = \text{P/As}$) display bands at 465 nm ($^1\text{A}_{1g} \rightarrow ^1\text{T}_{1g}$), 430 nm ($\text{T}_{2g} \rightarrow \pi^*$, LMCT), 340 nm ($n \rightarrow \pi^*$, LC), 292 nm ($\pi \rightarrow \pi^*$, LC) similar to those observed for the low-spin d^6 (Ru^{++}) complexes containing N, S – chelating thioamide, $\text{P}\phi_3$ or $\text{As}\phi_3$, and chloride ligands.²⁴⁻²⁶

The magnetic moment of ruthenium (III) complexes are found between 1.85 – 1.92 BM corresponding to one unpaired electron in T_{2g}^5 (Ru^{+++}) in octahedral structure.²⁷ The ground state of Ru (III) in octahedral environment is $^2\text{T}_{2g}$ arising from the $t_{2g}^5e_g^0$ configuration. Hence, two bands corresponding to $^2\text{T}_{2g} \rightarrow ^2\text{A}_{2g}$ and $^2\text{T}_{2g} \rightarrow ^2\text{T}_{1g}$ are possible. All the complexes display bands at 630 – 640 nm ($^2\text{T}_{2g} \rightarrow ^2\text{A}_{2g}$), 460-462 (LMCT) and 305 nm ($\pi \rightarrow \pi^*$) which indicates octahedral environment around ruthenium (III) ion and consistent to the other ruthenium (III) octahedral complexes.²⁸

Table – 1 : Analytical and Physical data of complexes

Complex ^ψ (Colour, M. Pt, 0°C)	μ _{eff.} (BM)	Analysis (%) : Found/(Calcd)				Molar cond. $\text{cm}^2\text{mol}^{-1}$
		C	H	N	Ru	
$[\text{RuCl}(\text{CO})(\text{P}\phi_3)_2\text{L}]$ (Greenish yellow, 157)	Diamag.	61.81 (60.69)	4.29 (4.28)	6.22 (6.15)	11.04 (11.10)	10.60
$[\text{RuCl}(\text{CO})(\text{As}\phi_3)_2\text{L}]$ (Yellow, 140)	Diamag.	55.21 (55.33)	3.99 (3.90)	5.46 (5.61)	10.10 (10.12)	15.60
$[\text{Ru}(\text{P}\phi_3)_2\text{L}_2]$ (Brown, 172)	Diamag.	60.88 (60.73)	4.50 (4.49)	10.50 (10.49)	9.50 (9.46)	10.20
$[\text{RuCl}(\text{P}\phi_3)_2\text{L}_2]$ (Green, 192)	1.85	51.40 (51.39)	4.01 (3.92)	13.22 (13.32)	12.32 (12.01)	8.90
$[\text{RuCl}(\text{As}\phi_3)_2\text{L}_2]$ (Green, 176)	1.83	49.01 (48.84)	3.88 (3.73)	12.62 (12.66)	11.50 (11.41)	12.60
$[\text{RuBr}(\text{P}\phi_3)_2\text{L}_2]$ (Yellowish green, 165)	1.92	48.88 (48.81)	3.80 (3.72)	12.66 (12.65)	11.50 (11.41)	11.65

Where, $\psi = \text{LH} = 1\text{-paraethoxyphenyltetrazoline-5-thione}$.

IR Spectra

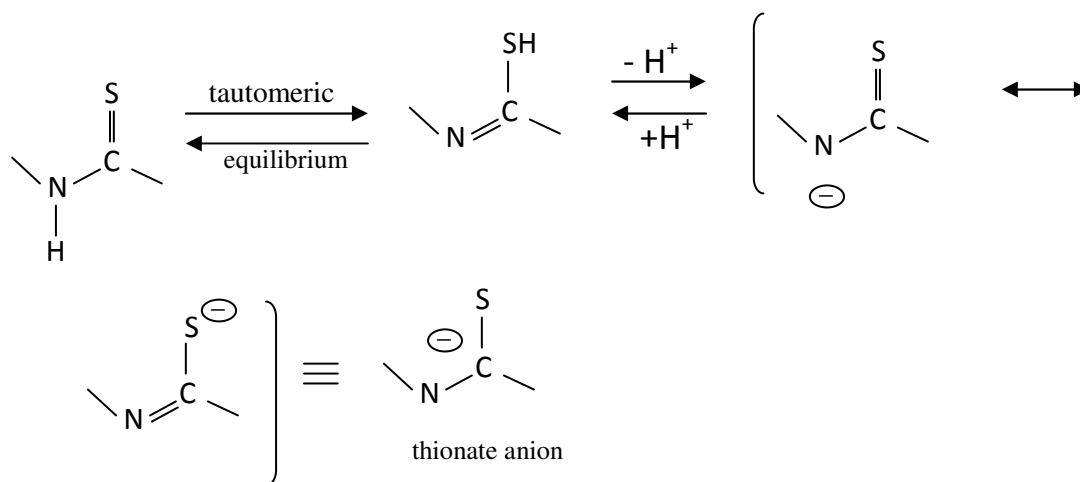
The thioamide ligand, 1-paraethoxy phenyl tetrazoline-5-thione exhibits characteristics bands νSH (2550 cm^{-1}), νNH (3150 cm^{-1}), thioamide band I (1515 cm^{-1}), band II (1288 cm^{-1}), band III (1060 cm^{-1}) and band IV (805 cm^{-1}) are affected on coordination with both Ru (II) and Ru (III) ion (table 2). The νSH and νNH bands of ligand are not observed in complexes indicating deprotonation and presence of

mononegative bidentate anion which enhances the donor capability of the sulphur atom via the N – to – S backbone.²⁹⁻³⁰

Table – 2 : Major IR Spectral bands (cm^{-1}) of ligand and complexes

Compounds	$\nu\text{N-H}$	Thioamide Bands*				$\nu\text{Ru-N}$ ($\nu\text{Ru-S}$)	$\nu\text{Ru-X}$	$\nu\text{Ru-E}$
		Band I	Band II	Band III	Band IV			
LH (ligand)	3150 (sb)	1515 (s)	1288 (s)	1060 (s)	805 (m)	-	-	-
$[\text{RuCl}(\text{CO})(\text{P}\phi_3)_2\text{L}]$	-	1490 (m)	1305 (m)	1020 (m)	760	480 m (410 w)	350 m	505 m
$[\text{RuCl}(\text{CO})(\text{As}\phi_3)_2\text{L}]$	-	1480 (s)	1310 (s)	1035 (m)	705 (m)	475 m (405 m)	350 (m)	495 m
$[\text{Ru}(\text{P}\phi_3)_2\text{L}_2]$	-	1495 (s)	1315 (s)	1030 (ms)	780 m	445 m (400 w)	-	500 m
$\text{RuCl}(\text{P}\phi_3)_2\text{L}_2]$	-	1480 (s)	1310 (s)	1035 (m)	785 m	475 m (405 w)	350 m	495 m
$[\text{RuCl}(\text{As}\phi_3)_2\text{L}_2]$	-	1490 (s)	1315 (s)	1030 (ms)	780 m	480 m (405 w)	355 m	490 m
$\text{RuBr}(\text{P}\phi_3)_2\text{L}_2]$	-	1480 (s)	1305 (s)	1025 (m)	790 (m)	485 m (400 w)	270 m	500 m

* Mixed Bands : Band I = $\delta\text{NH} + \delta\text{CH} + \nu\text{C}=\text{N}$; Band II = $\nu\text{C} - \text{N} + \delta\text{NH} + \delta\text{CH} + \nu\text{CS}$; Band III = $\nu\text{C} - \text{N} + \nu\text{C} - \text{S}$; Band IV = $\nu\text{C} - \text{S}$; E = P/As; X = Cl/Br



Scheme-1

The formation Ru – N and Ru – S bond is confirmed by the change in position and intensity of thioamide bands on complexation.³¹ The thioamide band II blue shift and thioamide band III and band IV red shift to lower wave number (table 2) indicating bonding of thioamide ligand through both N and S considering our previous observations.³²⁻³⁴ New bands around 530, 690, 740 and 1560 cm^{-1} due to coordinated $\text{As}\phi_3$ and near 390, 430, 505, 1090 cm^{-1} due to coordinated $\text{P}\phi_3$ are agreement with previous literature.³⁵⁻³⁶ New strong band in the region $1980 - 1985 \text{ cm}^{-1}$ due to terminally coordinated carbonyl group is at higher frequency than in the precursor complexes is also consistent with previous literature.³⁷ Such band is not present in non-carbonyl complexes. The presence of single Ru – P stretching mode in $[\text{Ru}(\text{P}\phi_3)_2\text{L}_2]$ indicates two $\text{P}\phi_3$ group at trans-position in octahedral structure.³⁸ Thus, the thioamide ligand acts as N-S – chelating mononegative bidentate nature in all octahedral complexes.

Table – 3 : Antibacterial activity of ligand and Ruthenium (III) chelates at different concentration (PPM)

Compounds	Diameter of inhibition (mm)								
	S. Aureus			B. Subtilis			E. Coli		
	25	50	100	25	50	100	25	50	100
LH (ligand)	-	+	++	-	-	+	-	-	+
RuCl(Pφ ₃)L ₂	+	++	++	-	+	++	-	+	++
[RuCl(Asφ ₃)L ₂]	+	++	+++	NT	NT	NT	+	++	+++
[RuBr(Pφ ₃)L ₂]	+	+	++	+	++	+++	NT	NT	NT
Streptomycin (stand.)	++	+++	+++	++	+++	++++	++	+++	++++

Inhibition diameter in mm : (+) 15-20 mm; (++) 20-25 mm; (+++) 25-30 mm; (++++) 30-35 mm; (-) inactive zone < 10 mm; NT = Not tested

Table – 4 : Antifungal (%) inhibition of ligand and ruthenium (III) complexes at different concentration (μg ml⁻¹)

Compounds	A. Flavus			C. albicans			A. Parasiticus		
	25	50	100	25	50	100	25	50	100
LH (ligand)	-	+	+	-	-	+	-	+	++
RuCl(Pφ ₃)L ₂	-	+	++	-	+	++	-	+	+
[RuCl(Asφ ₃)L ₂]	+	++	++	+	++	+++	+	++	++
Grisofulvin (stand.)	++	+++	++++	NT	NT	NT	++	+++	++++

Inhibition diameter in mm : (+) 15-20 mm; (++) 20-25 mm; (+++) 25-30 mm; (++++) 30-35 mm; (-) inactive zone < 10 mm; NT = Not tested

¹H NMR Spectra

¹H NMR spectra of thioamide ligand (LH), precursor complex, [RuH(CO)(Pφ₃)₃Cl] and [RuCl(CO)(Pφ₃)₂L] were recorded in CDCl₃/TMS to substantiate further mode of metal-ligand bonding. The precursor complex exhibit signal in the δ8.13 – 8.80 PPM range due to aromatic proton of Pφ₃ molecule.³⁹ However, thioamide derivative of precursor exhibits broad multiplet in the region δ7.55 to δ7.72 PPM due to phenyl protons. The broad nature of peak may be probably due to larger quadrupole resonance broadening effect of tetrazole nitrogen atoms or ligand exchange reaction occurring in solution. The imino proton of thioamide ligand observed at 1.22 PPM was found to be absent in the spectrum of [RuCl(CO)(Pφ₃)₂L] indicating deprotonation⁴⁰ during complex formation consistent with the results of IR spectra.

Antibacterial Activity

The in vitro antibacterial screening of ligand and its ruthenium (III) chelates have been carried out against S. Aureus, B. Subtilis and E. Coli using a nutrient agar medium by Disc diffusion method.⁴¹ The complexes to be tested were dissolved in DMSO to a final concentration of 0.25%, 0.5% and 1% and soaked in filter paper disc of 5 mm diameter and of 1 mm thickness. The discs were placed on the previously seeded plate and incubated at room temperature for 24 h. The diameter of inhibitory zone around each disc was measured. Streptomycin was used as standard.

The results (table-3) showed that the complexes exhibit moderate activity and the toxicity of ruthenium chelates increases on increasing the concentration.⁴² Metal chelates are more active than thioamide ligand.

Antifungal Activity

The thioamide ligand and its ruthenium (III) chelates were screened against A. Flavus, A. Parasiticus and C. Albicans using cup-plate method reported in literature⁴³ using DMSO as solvent. The inhibition zone

formed around each filter paper was measured. The standard fungicide carbendazim was used for comparison and the results are given in table 4. The antifungal activity was more confined with the metal chelates containing triphenyl arsine and negligible activity against these organism was observed at lower concentration.

ACKNOWLEDGMENTS

One of the authors (KVG) thanks UGC, New Delhi to award Junior Research Fellowship (JRF, NET) and Principal Dr. P.K. Verma for providing facilities.

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[RJC-1107/2014]

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