International Journal of Research
Available at https://edupediapublications.org/journals
p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 08
July 2017

Co-Ordination Behavior of Amino Thiozole with Transition Metal Complexes

Neetu Gaur
Assistant Professor
Department of Chemistry
Parishkar College of Global Excellence
Jaipur

ABSTRACT

The ambidentate behaviour of diazoles is well known - The substitution of an amino group makes the ligand more efficient coordination due to increase in one more binding site. In addition the substitution at 2 position makes the ligand more suitable to act as a chelating agent. Therefore it promoted us to study the coordination behavior of 2 amino-diazole towards some metals of the first transition reactions with other ligands such neutral ligands like pyridine, picoline etc will be. studies. Complexes of 2 amino diazoles with the first sow transition elements salts and the ligand in appropriate solvents. The solvents will be aried by standard method (36) the metal will be estimated by the usual procedure (36). Sulpher and halogens will be estimated by fusion method and nitrogen by kjeldahi's method the complex prepared will be structurally investigated by infrared and electronic spectra.

INTRODUCTION

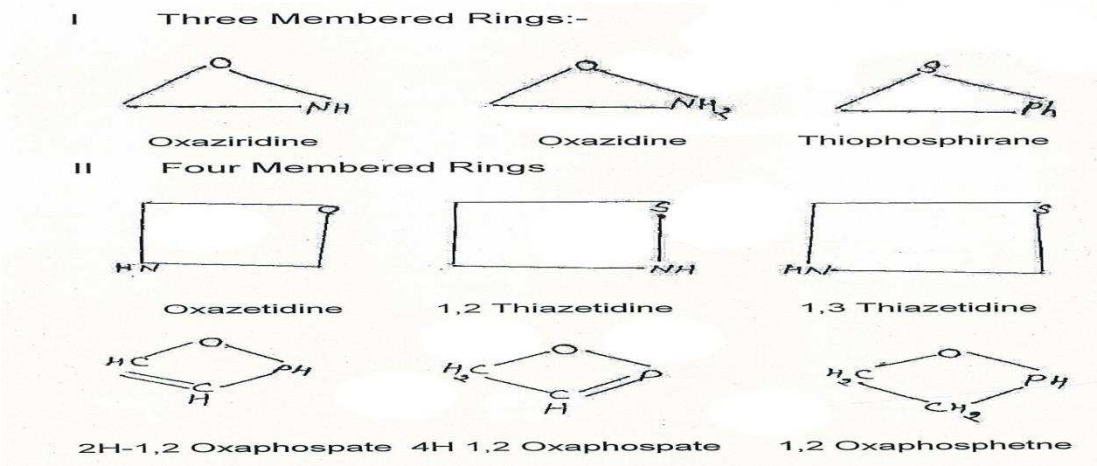
Heterocyclic compounds containing two hetero atoms our abundantly found in biological systems. Numerous azoles like imidazoles, indazoles and thiazoles. The recent research has shown that thiazole antibiotics cancer comes and inhibit a protien called fox m^1. It is suggest that the drugs ability to inhibit fox m1 may account for their anti - cancer activity fox m^1 is one of the most highly produced proteins in cancer ails and in believed to play an important role in causing ails to become cancerous. Because production of the protein is not usually switched. On is non dividing cells, the protein may present a promising target for anticancer treatments. It had also been found that these thiazole antibiotics actually attacks other protein involved in cancer and stabilized the cancer causing protien (3). In addition to their biological significance, the polyheterocyclic compounds constitute a vast storehouse of ligands with potential ambidentate character and to the presence of different hetero atoms. Substitution at different positions in these polyheterocyclic ring system affects their chemical as well as their coordination behaviour.Very often it has been found that even the mode of linkage with the metal atoms may be affected (4,5) In the presence of sub subsituted containing donor atoms, the co-ordination modes of heterocyclic compounds become further interesting.

As the group V and VI elements are good donors, a very large no of heterocyclic compounds containing group V and group VI Clements as heteroatom have been used as ligands. Therefore it was thought interesting to study the co-ordination behaviour of azoles containing one group V heteroatom's as nitrogen and other group VI hetero atom as sulpher i.e. thiazole. The important classes of polyheterocyclic ring system containing a group V and

p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 08
July 2017

group VI hetero atoms are given below. The system containing more than two hetero atoms have not been included due to limitation of space.

RING SYSTEM CONTAINING A GROUP V AND A GROUP VI HETERO ATOMS



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Volume 04 Issue 08
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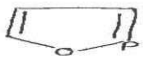
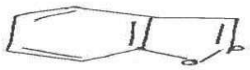
III Five Membered Rings :-

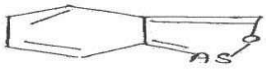


1, 2 - Oxaphosphote



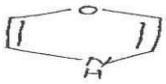
1,2 - benzoxaphosphole

1,2- Oxarsole



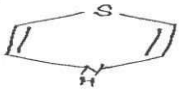
2,1 - Benzoarsenole

IV Six Membered Rings :-

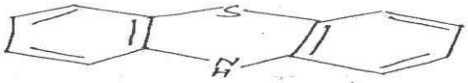


1,4-Oxazine

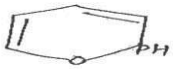
Phenoxazine



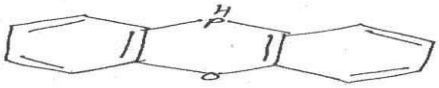
1,4 -Thiazine



Pherothiazinl

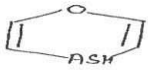


2H-1,2 Oxaphosphorin

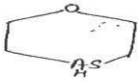


Phenoxaphosphinl

Phenothiaphosphine



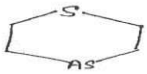
4H-14, - Oxarsenine



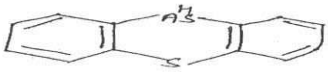
1,4-Oxarsone

Phenoxarsine

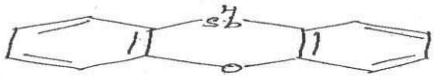
4H-1,4 Thair



1,4 - Thiarsenane



Phenothiarsive



(Phenoxartimonin)

p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 08
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THIAZOLES

(Molecular and Electronic Structure)

Thiazoles are members of the azoles hiterocyles that includes imidazoles and oxazoles, thiazole can also be considered a functional group oxazoles are related compounds, with sulfur replaced by oxygen. Thiazoles are structurally similar to imidozoles, with the thiazole sulpher replaced by nitrogen.Thiazole rings are planar and aromatic thiazoles are characterized by larger pi-electron delocalization than the corresponding oxazoles and have therefore greater aromaticity. This aromaticity is evidenced by the chemical shift of the ring protons in proton NMR spectroscopy (between 7.27 and 8.77 PPM), clearly indicating a strong diamagnetic ring current. The calculated pi-electron density makes Cs as the primary site for electrophillic substitution, and C_2 as site for nucleophillc substitution.

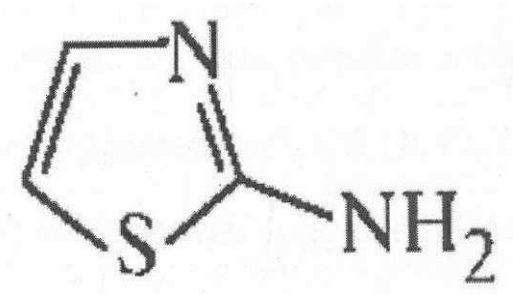
(Occurrence of thizloes and thiazolium salt):

Thiazoles are found is a variety of specialized product often fused with benzene derivatives, the So-called benzothiazoles. In addition to vitamin B1, the thiazole ring is found to epothilone, other important thiazole derivative are benzothiazoles for eg: - the firely chemical luciferin. Whereas thiazoles are are well represented in biomolecules oxazoles are not.

COMPLEXES OF 2-AMINOTHIAZOLES

Compounds of thiazoles with several transition elements egCo(ll), Ni (II), Cu (II), Cu(ll), ln(lI) Pd(ll) and Rh(lll) etc. have been isolated. The substituted thiazoles like 4-methyl thiazole, 2,4- diethyl othiazole, 2, ethyl -4 methyl thiazole, 2-hydroxyl methyl thiazole have also been exploited to study their co-ordination mode towards several Transition Elements.-

The substation of an amino group makes the ligand more efficient for co-ordination due to increase in one more binding site in addition, the substitution at 2 position makes the ligand more suitable to out as a chelating agent. Therefore it prompted us to study the coordination behavior of 2 -aminothiazole towards. Some metals of the first transition series and if possible the substitution reaction with other ligands such as pseudohalides eg:SCN and also neutral ligands like pyridine, picoline etc. will be studied.



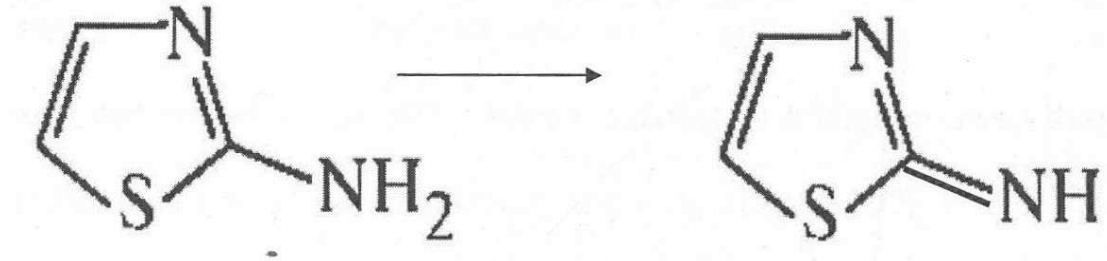
2-aminothiazoles has a great potentiality to act as an ambidentate ligand. Moreover the substitution of an amino group at 2-position makes the ligand more versatile. The ambidentate behaviour of the ligand can be shown as:

(i) The ring nitrogen versus ring sulphur atom.

(ii) The ring nitrogen versus exo-cyclic nitrogen atom.

(iii) The ring sulphur versus the amino nitrogen linkage and

(iv) Due to the possibility of tautomerisation the ligand may act as a chelating agent as well.



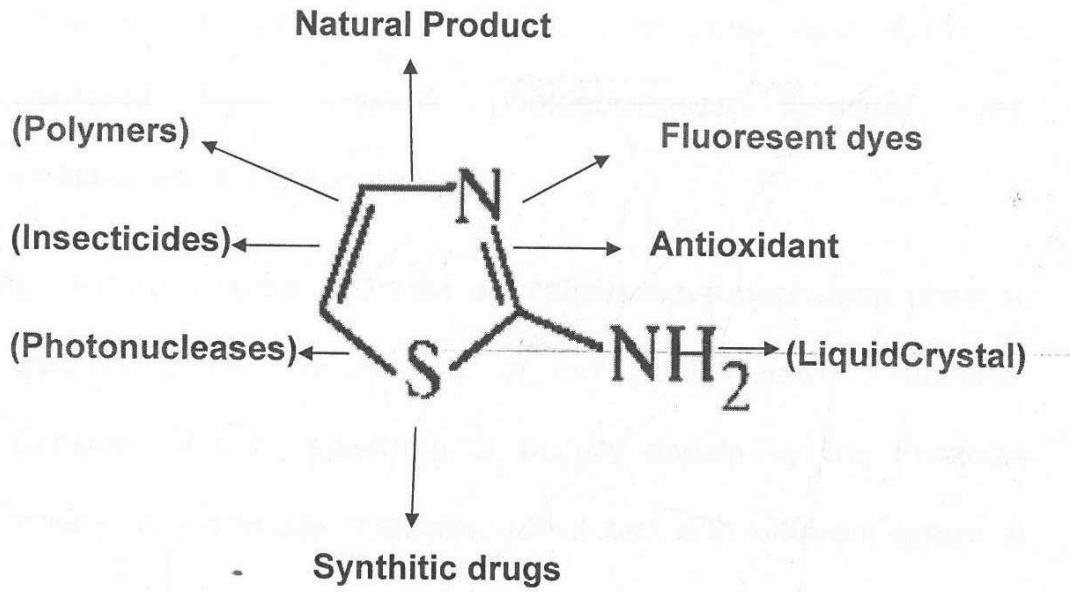
In the complex of 2-amino thiazole with first second and third series transition Clements. Various possible modes of linkage of the ligand have been suggested In Ti, Co, Ni, Pt, Cu. In compexes, bonding through ring nitrogen was suggested. Howeyer in some of the Mn, Cc, Ni and Cu complexes it was concluded that the ligand is amino nitrogen bonded (13) through it may also be sulpher bonded in Cn (II), Cd (II) and Hg (Il) (13).

The complexes were also shown to have different gcometries like tetrahadral, octahadral at well as the present of ligand in bridge position from the above discussion, it is clear that the actual structure of 2 - aminothiqzole compexes is very likely influenced by the nature of the ligand as well as the central metal ion. There for

to study the bonding mode of 2 - aminóthiazole with first row transition metals spicially cobalt (II) forms the subject matter of this discussion.

2- aminothiazoles derivative were first exported by Hantzsch and swber in 1887. The importantance of 2 - amino thiazole ring sys was enhaneed in the 1930. When williams and dine showed that (bitamin B,) contained a thiazole ring and latter in 1939 one of the major sulfa drugs, Sulfathiazoles was produced.The class of heterocyclic compound known as 2 - aminothiazole is found in many natural and synthitic product with wide range of pharmacological activity. In the case of natural product 2 - aminothiazole is present as a subunit in a large no. of tessestrial and marine compound with different biological activities that represent a very important field in drug discovery.The depiction of various application of 2 - aminothiazoles is described in figure due to importance of this molecule many scientist have focused their research on 2 -

amino thiazoles. Commercial significance thiazoles include mainly dyes and fungicides.



2-amino thiazoles are widely used as accelerators in rubber vulcanization and as antioxidants. A large no of dyes are derived from thiazoloium salt.

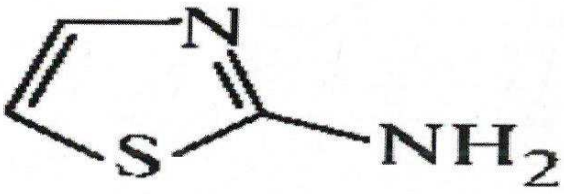
2-Amino thiazoles have been used in varieties of applications, which covers the field of agriculture, pharmacy, photography or related activities various thiazoles derivatives have found to posses antibacteria/ fungicides, antiinflammatory, antihalminties, antitubercular, anti HIV, Herebicides etc. They are also among one of the key building blocks in drug discovery that can be well illustrated by large no of drugs in the market. Containing this functional group.Thiazole ring also find applications in other field. Such as polymers, liquid crystals, photonucleases, florecent dyes, insectisides and antioxidant.

2 - Amino thiazole and their derivatives have been long used as precussors for the synthesis of biologically active molecules, Because of wide spectrum of activity shown by the thiazoles moiety, numberoues thiazoles subsituted with different group at various positions have been prepared.

2 - Amino - 4 substituted and thiazoles in the presence of various reagents, undergo different types of reactions to field other heterocyelic compounds known as synthetic intermidiates and therapeutic agents s-alkyl 2 - Phenylalky, carbonyl amino 1,3 thiazoles are known as protein kinase inhibitors.

REVIEW OF LITERATURE

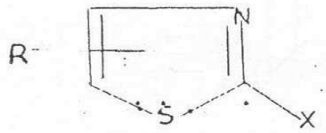
A review relating to the coordination behaviour of thiazole and 2 - aminothiazole is very important as it will help in the methods of synthesis of complexes as well as in indicating the mode of bonding in the complexes synthisized by us. Therefore, in the following discussion the various complexes synthesized by thiazole are given alongwith the special features wherever they are given - The complexes of thaizole are present in the following Table



D THIAZOLES :

(i) Complexes of thiazole and its derivative

Table - 7



(XIX)

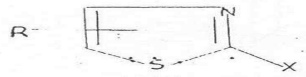
X	R	M	Complexes	Sterochem. & linkage	Special features	Ref.
		V^{III}	M:L=4:1	A	A	184
			$[V_2(L)_6Cl_5]^+$	A	A	11
		Cr^0, Mo^0, W^0	$M(CO)_5L$	M-N	Mass spectral study	185
			$M(CO)_{6-x}L_x$ (x=1 2,3)	A	Thermochem. study	186 187
			$M(CO)_5L$	Oct.	Electronic MCD spectra	188
		Tc^V	$TcO(OR)Cl_2$	Dist.oct	A	189
		Fe^{II}	[Fe(PPDME) (NO)L]	A	EPR study	190,191
		Ru^{IV}	No Complex	A	Ru estimation	192
		Co^{II}	$CoLX_2$(X=Cl,Br)	Oct.	A	193,194A
			CoL_2X_2(X=Cl,Br)	Oct.(halide bridg.	A	"
			CoL_2I_2	Tet.,M-N	A	"
			CoL_4X_2(X=Br I,SCN)	Oct.,M-N	A	194A
			$CoL_6(ClO_4)_2$	Oct.,M-N	A	194A
			$Et_4N(CoLBr_3)$	Tet.	A	194A
			$CoM(NCS)_4nL$	Oct.,bridg.	A	195
			M=Zn,Cd,Hg n=2,4 or 6	NCS		

Contd.

D THIAZOLES :

(i) Complexes of thiazole and its derivative

Table - 7



(XIX)

X	R	M	Complexes	Sterochem. & linkage	Special features	Ref.
		V^{III}	M:L=4:1	A	A	184
			$[V_2(L)_6Cl_5]^+$	A	A	11
		Cr^0, Mo^0, W^0	$M(CO)_5L$	M-N	Mass spectral study	185
			$M(CO)_{6-x}L_x$ (x=1 2,3)	A	Thermochem. study	186 187
			$M(CO)_5L$	Oct.	Electronic MCD spectra	188
		Tc^V	$TcO(OR)Cl_2$	Dist.oct	A	189
		Fe^{II}	[Fe(PPDME)(NO)L]	A	EPR study	190,191
		Ru^{IV}	No Complex	A	Ru estimation	192
		Co^{II}	$CoLX_2$ (X=Cl,Br)	Oct.	A	193,194A
			CoL_2X_2 (X=Cl,Br)	Oct.(halide bridg.	A	"
			CoL_2I_2	Tet.,M-N	A	"
			CoL_4X_2 (X=Br I,SCN)	Oct.,M-N	A	194A
			$CoL_6(ClO_4)_2$	Oct.,M-N	A	194A
			$Et_4N(CoLBr_3)$	Tet.	A	194A
			$CoM(NCS)_4nL$ M=Zn,Cd,Hg n=2,4 or 6	Oct.,bridg. NCS	A	195

Contd.

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Available at https://edupediapublications.org/journals
p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 08
July 2017

	$[LCo\{(DO)(DOH)pn\}**CH_3]X$		NMR study		196
	$(X=ClO_4, PF_6)$	A			
Co^{III}	Pentammine Cobalt(III)Complex	Co-N	Kinetic study		197
Rh^{I}	$[Rh(CO)_2LCl]$	A	Antitumour		198
Rh^{III}	$[RhL_4Cl_2]Cl.5H_2O$	Oct.,M-N	Antibacterial, antitumour		199,200
	$[FhL_4Br_2]Br.2H_2O$	Oct.,M-N	"		"
	$[RhL_4Cl_2]Cl$	A	Biol.activity		201,202
	Rh^{III} complex	A	Antitrypanosomal		203
Ni^{II}	NiL_2X_2	Oct.,anion bridg.,M-N	A		194
	$(X=Cl, Br, SCN)$				
	NiL_4X_2	Oct.,M-N	A		193,194
	$(X=Cl, Br, I, SCN, ClO_4)$				
	$NiL_6(ClO_4)_2$	Oct.,M-N	A		194
Pd^{II}	PdL_2X_2 $(X=Cl, Br)$	Sq-planar, M-N	A		204
Pt^{II}	PtL_2Cl_2	"	A		205
	cis PtL_2Cl_2	A	Antitumour, antibacterial		206
	cis $Pt(NH_3)LCl_2$	M-N(ring)	Cytotoxic		95
Pt^{IV}	$PtL_2Cl_2(OH)_2$	A	Antitumour antibacterial		206
Cu^{II}	$CuLCl_2$	Sq-planar	A		194
	CuL_2X_2 $(X=Cl, Br)$	Dist.oct., M-N	A		207, 208
	CuL_4X_2 $(X=NO_3, ClO_4)$	Oct.	A		194
	$CuL(OAc)_2$	A	Antiferr.		209

	Cu^{II}	CuL_2Cl_2	Ploy.halide bridg.	A	207
	Ag^{I}	Silver complex		Stability const.	210
	Au^{III}	no definite complex		Au determinatn. by titratn.	171
	Zn^{II}	ZnL_2X_2 $(X=Cl, Br)$	Tet.	Stability const.	172
		ZnL_4X_2 $(X=NO_3, ClO_4)$	A	"	172
	Al^{III}	$AlBr_3L$	M-N	Heat of formatn.	194
		$2AlBr_3.L$	M-N; M-π (ring)	"	
		Al^{III}, Ga^{III} & In^{III} halide complexes	A	A	194
	Sb^{V}	$SbCl_5L$	M-N	A	211
Br	H	Co^{II}	CoL_2X_2	Tet.,M-N	A	212
		Pt^{II}	cis $Pt(NH_3)LCl_2$	M-N(ring)	Cytotoxic	95
		Cu^{II}	CuL_2X_2 $(X=Cl, Br)$	Sq-planar, M-N	A	212
Br	5-NO_2	Pt^{II}	PtL_2X_2 $(X=Cl, I)$	A	Antitumour	213
		Rh^{I}	$[Rh(CO)_2LCl]$	A	"	190
		Rh^{III}	$[RhL_4Cl_2]Cl$	A	"	214,202
Me	H	Ag^{I}	Silver complex	M-L_π bonding	A	215
		Al^{III}	Chloro complex	A	NMR study	216
		Eu	Europium complex	A	A	215
H	4-Me	Co^{II}	CoL_2X_2 $(X=Cl, Br)$	Tet.	S	205,217,218

Contd.

International Journal of Research
Available at https://edupediapublications.org/journals
p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 08
July 2017

	Ni^{II}	NiL_2X_2 (X=Cl,Br)	Oct., x-bridg.	S		205,217,218
	Pd^{II}	trans-PdL_2X_2 (X=Cl,Br)	Sq-planar, M-N	A		204,218
	Pt^{II}	cis-PtL_2X_2 (X=Cl,Br)	Sq-planar, M-N	A		205
	Cu^{II}	CuL_2X_2 (X-Cl,Br)	Pseudo Oct., x-bridg	S		205,217,218
		$CuL\ Cl_2$	X-bridge	A		218
		$[CuL_2Br_2]_2$	Teg.-py	X-ray	study	219
		CuL_3Cl_2	A	A		220,221
		$[CuL_2Cl_2]_2$	Cu^{I} pseudo tet., Cu^{II}	A		221
		$[CuL_2Cl_2]_2$.MeOH	Dist.teg-py	A		221
		$[CuL_2Cl_2]_2$.MeOH	"	X-ray	study	220
	Cu^{II-I}	$Cu_2L_4Cl_3$	X-bridg.	A		221
	Cu^{II}	$[CuL(DMF)Cl_2]_2$	Dist.teg-py	X-ray	study	222
	Ag^{I}	Ag complex	A	S		215,31
	Zn^{II}	ZnL_2X_2 (X=Cl,Br)	Tet.	S		205,216
		Eu complex	A	A		223
H	5-Me	"	A	A		223
Me	4-Me	Co^{II} CoL_2X_2 (X=Cl,Br)	Tet.,M-N	S		205,223
		Rh^{III} $[RhL_4Cl_2]Cl$	A	Antitumour		202
		Ni^{II} NiL_2X_2 (X=Cl,Br)	Sq-planar, M-N	S		205,224
		$NiM(NCS)_4.nL$ (M=Zn,Cd,Hg; n=2, 4 or 6)	A	A		225

Contd.

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			$[NiL_4]_4[Zn(NCS)_4]$	A	A	225
			$[L_4Ni(NCS)_2]$	Monomeric	A	225
			$[Cd(SCN)_2]$	bridg.		
			$[L_2Ni(NCS)_2]$	Polym.bridg.	A	225
			$[Hg(SCN)_2]$			
		Cu^{II}	CuL_2X_2	Sq-planar	S	205,224,226
			(X=Cl,Br)	M-N		
			$CuL(OAc)_2$	A	Antitumour	227
Me	5-Me	Rh^{III}	RhL_4Cl_2Cl	A	Antitumour	202
	5-Cl	Rh^{III}	RhL_4Cl_2Cl	A	"	202
Et	4-Me	Co^{II}	CoL_2X_2(X=Cl,Br)	Tet.,M-N	A	205
		Ni^{II}	NiL_2X_2(X=Cl,Br)	Sq-planar,M-N	A	205
Et	H	Eu	Europium complex	A	A	223
Iso-pr	H	Pt^{II}	cis PtL_2I_2	A	Antitumour antitrypanosomal	222
		Pt^{IV}	$PtL_2I_2(OH)_2$	A	"	222
1-Me	H	Rh^{I}	$[Rh(CO)_2LCl]$	A	Antitumour	198
		Rh^{III}	$[RhL_4Cl_2]Cl$	A	Antibacterial antitumour	201, 202
		Pt^{II}	cis PtL_2Cl_2	A	Antibacterial antitumour	203, 204
		Pt^{IV}	$PtL_2Cl_2(COH)_2$	A	"	"
			Eu complex	A	A	223
1,1-Di-MeEt	H		Eu complex	A	A	223
Ph	H	Pd^{II}	Mononuclear cycle Pd complex	A	A	228

Contd.

Al^{III}	Chloro complex	A	A	216, 229

** (DO)(DOH)pn = N^2, $N^{2'}$ - propanediylbis (2,3 butane dione -2-imino-3-oxime

* PPDME = protoporphyrin dimethyl ester.

(ii) 2-Thiazolamine -

Complexes of 2-thiazolamine with the metal ions Ti^{IV} (230, 231), Mn^{II}(232), Co^{II}(212,232-38), Co^{III}(239-41,242), Rh^{I}(198), Rh^{III}(201,214,243), Ni^{II}(212,232,234,236,244,245), Pd^{II}(246-48), Pt^{II}(214,243,246-48), Cu^{II}(212,236,244,249,250,113,150), Ag^{I}(250A, 30), Zn^{II}(232,234,242,251), Cd^{II}(232,234,244), Hg^{II}(234, 244), Eu^{III}(58), Tm^{III}(58), Yb^{III}(58) Si^{IV}(223), Ge^{IV}(252), Sn^{IV}(230, 252-55) and Te^{II}(256) have been reported.

Ti^{IV} complexes, TiL_2X_4(X=Cl,Br,I) are octahedral with cis-stereochemistry and N-bonded ligand (230). Some mixed ligand complexes containing 2-thiazolamine and sulpha drugs have also been reported (231).

Duff et al (212) prepared complexes, CoL_2X_2(X=Cl,Br,I) and CoL_4X_2(X=Cl,Br), CoL_2X_2(X=Cl,Br,I). (233) and X=SCN were also reported by other school of workers. Duff et al reported the bonding through ring nitrogen (212), however Singh and Srivastava(233) suggested amino nitrogen bonded on the basis of spectral studies for the halogeno complexes and Singh and Shukla (234), proposed S-bonded ligand in $CoL_2(SCN)_2$. Raper et al (235), showed form x-ray structural studies that in CoL_2Cl_2, the metal atom is tetrahedrally coordinated to the Cl-atoms and the heterocyclic

N-atom in the thiazole ring; the amino groups of the ligands form both intra and intermolecular hydrogen bonds to the chlorine atoms. The thermal decomposition of CoL_2X_2(X=Cl,Br) have been studied and the end products identified by x-ray diffraction(236). From thermal and x-ray analysis a compound $CoL_4(NCS)_2$ was characterized and show to contain $Co-N_6$ octahedral. The infrared and electronic spectra are also consistent with N-donation by both ligands. The complex autocatalytically decomposed on heating in nitrogen (237). The amino bonded bimetalic complexes of Co^{II} and Ag^{I} or Cu^{II} having the composition $Co[Ag(XCN)_2]_2.6L$(X=S,Se) and $Co[Cu(SCN)_2]_2.6L$ (238) were prepared; their electronic spectra and magnetic moments suggests octahedral stereochemistry, and the molar conductance indicate their cationic-anionic nature.

Oxidation of Co^{II} salts in the presence of ethylenediimino bisacetylacetone (H_2Q) and tertiary amines led to the formation of CoL_2QX(X=Cl,Br,I), $\frac{1}{3}[Cr(NCS)_6]^{3-}$, $[Cr(NCS)_4(PhNH_2)_2]^-$. With 1,2-propanediiminobisacetylacetone (H_2Q'), $CoL_2Q'X$ for X=I, $1/3[Cr(NCS)_6]^{3-}$ $[Cr(NCS)_4(PhNH_2)_2]^-$, $[Cr(NCS)_4(NH_3)_2]^-$ were also prepared and characterised by infrared and electronic spectra and thermal analytical methods (239). The Co^{III} complexes CoL_2Q_2X (Q=4-methyl-2,3-pentanedionedioime (propoxime), (X=Br,I) have also been reported (240). The kinetics of solvolysis of cis-$[Co^{III}BrL(en)_2]^{2+}$ cation were investigated (241,242).

Rh^{III} and Pt^{II} complexes of the type $[RhL_2X_2.OH.H_2O].C_2H_5OH$(X=Cl,Br,I) and $[Pt\ LClOH.H_2O].C_2H_5OH$ were shown to have

S-bonded ligand (243). $[Rh(CO)_2LCl]$ was examined for its antitumour activity (198). The isolation and study of antitumour activity of $[RhL_4Cl_2]$ Cl have also been reported (201, 214).

Duff et al (212) first isolated NiL_4X_2(X=Cl,Br,SCN) and $NiLCl_2$. Manhas and Bhatia (244) and Singh etal (234,245) also studied NiL_4X_2(X=Cl,Br,I,ClO_4) (245) (X=SCN) (234) and (X=OAc) (244). Jordan et al investigated the thermal decomposition of the complexes NiL_4X_2(X=Cl,Br) (236). Singh and Srivastava (245) prepared a complex $L_2[NiL_4(ClO_4)_2]$. NiL_4X_2(X=Cl,Br) (212,234,244, 245) and $NiLCl_2$ were shown to have distorted octahedral geometry. In NiL_4Cl_2, the ligand is bonded through the ring nitrogen and for $NiLCl_2$ polymeric octahedral structure involving both chlorine and ligand bridged are suggested on the basis of electronic spectral data (212).

Cis-PtL_2X_2(X=halogen) were synthesized and shown to have antitumor activity (214). In acidic medium, 2-thiazolamine forms $Bu_4N[PdCl_3L]$, trans-PdL_2Cl_2 and cis-PtL_2Cl_2 (246) in which the ligand is bonded through ring nitrogen. In alkaline medium, ring cleavage of the coordinated ligand leads to monomeric $PdL_2'Cl_2$ and dimeric $[ML_2'Cl_2]_2$ (M=Pd,Pt; L'=HS.CH:CH.NH.C:N) (247). Stereo-chemistry, vibrational, UV and NMR spetra of the Pd^{II} and Pt^{II} complexes are discussed in relation to their biological activities(248)

Copper (II) complexes, CuL_4X_2[(X=Cl) (212,244), Br(212)], CuL_2Cl_2(212) and $CuL(OAc)_2$ (244) have distorted octahedral geometry. The thermal decompositon behaviour of CuL_2X_2(X=Cl,Br)

p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 08
July 2017

has been studied (236). The ligand is proposed to be coordinated via ring nitrogen in these complexes (249). Gamovskii et al also proposed the linkage of the ligand via endocyclic nitrogen in $CuL(OAc)_2$, $CuL_2(OAc)_2$ (150), and $CuL_2(OAc)_4$ (113) and attributed the decrease of NH_2 group valance vibrational frequency observed in these complexes to the inter-or intramolecular hydrogen bonding. X-ray crystal structure determination of 2-amino-2-thiazolium tetrachlorocuprate suggested the presence of distor ted $CuCl_4^{2-}$ tetrahedral and the protonation of ring nitrogen rather than amino nitrogen (250) from the solution studies of Mn, Co, Ni and Cu complexes, it was concluded that the ligand is amino nitrogen bonded in all cases though, it may be S-bonded in the copper complexes (232). Complexation of 2-thiazolamine with $AgNO_3$ in DMSO was indicated by ^{13}C and ^{109}Ag NMR chemical shifts studies (250A). Stability constant of Ag^I complex has been measured in aqueous solution (30).

2-Thiazolamine forms complexes, $ML_2(SCN)_2$(M=Zn,Cd,Hg)(234) and $CdLCl_2$ (244). The ligand is bonded through ring nitrogen in Zn^{II} and sulphur in Cd^{II} and Hg^{II} complexes (234); no comment was made on the bonding of the ligand in the chloro complex of cadmium (244). ZnL_2X_2(X=Cl,Br,I) were isolated and tested for their herbicidal and growth regulating activities (251). From solution studies, Zn^{II} and Cd^{II} complexes are, however, indicated to be amino nitrogen bonded (232).

Chlorosulphates of Eu^{III}, Tm^{III} and Yb^{III}, $M(SO_3Cl)_3$ form complexes with 2- thiazolamine in DMF, which were characterized by

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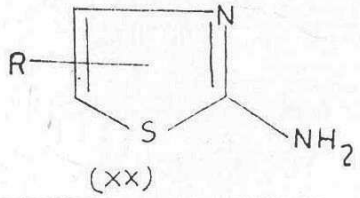
p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 08
July 2017

elemental analysis and the infrared data M $(SO_3Cl)_3L_2$ (M=Eu, Tm, Yb) (231).

SiF_4L was isolated and shown to contain both exocyclic and endocyclic nitrogen atoms linked to silicon (223). In the Ge^{IV} complexes, GeL_2X_4(X=Cl,Br) (252) and the Sn^{IV} complexes, SnL_4X_4(X=Cl,Br) (230,252), I(230), the ligand is bonded through the amino nitrogen. Addition compounds $Ph_3SnNCX.L$(X=O,S) having 1:1 stoichiometry were isolated (253,254), in which the infrared spectra indicated M-S linkage. Adduct of Ph_3SnN_3 with the ligand has also been prepared and shown to have M-N(NH_2) linkage (255). The interaction of organotellurium trichlorides p-$RC_6H_4TeCl_3$(R=H, OH, OCH_3,OC_2H_5) with the ligand led to the isolation of stable molecular adducts which were found to be more active against bacteria than fungi (256).

Several derivatives of 2 thiazolamine (XX) function as ligands and their complexes are summarised in Table-8.

Table-8 Derivatives of 2-thiazolamine and its complexes



(XX)

R	M	Complexes	Stereochem. & linkage	Remarks	Ref.
4-Cl	Pt^{II}	PtL_2I_2	-	Antitumor antitrypanosomal	222

Contd.

	Pt^{IV}	$PtL_2I_2(OH)_2$		Antitumor antitrypano-somal	222
5-Br	Rh^{III}	$[RhL_4Cl_2]Cl$	A	Antibact. & antitumour	201
	Pt^{II}	$cis-PtL_2Cl_2$	A	Antitumour	206
	Pt^{IV}	$PtL_2Cl_2(OH)_2$	A	"	206
$5-NO_2$	Rh^{III}	$[RhL_4Cl_2]Cl$	A	Antitumour	202
	Pd^{II}	$trans-PdL_2Cl_2$	Sq-planar, M-N	Radiosensi tive drug	257
		$[PdL_2]Cl_2$	Sq-palanr $M-N(NH_2)$	Radiosen-sitive drug	257
	Pt^{II}	$PtL_2X_2(X=Cl,I)$		Antitumour	213,258
		$[PtCl_2(NH_3)L]$	A	"	109
		$[PtCl_2L_2]$	A	"	109
		PtL_2Cl_2	A	Cytotoxic	108
		ML_2Cl_2(M=Co,Ni, Cu,Cd), NiL_3Br_2. MeOH, $NiL_2(NCS)_2$, $NiL_2(NCS)_2$.MeOH $(MeOH)_2$, AgL_2NO_3. $CuL(OAc)_2$ & CuL_2Cl	M-N	A	259
		$ML_2(SO_4)(DMF)_2$ (M-Co,Cu), NiL_2Cl_2 $(DMF)_2$, $CuL(SO_4)$ (DMF) & $CuLCl_2$ (DMF)	$M-N(NH_2)$	A	259
	Si^{IV}	$SiF_4 2L$	A	A	260
4-Me	Mn^{II}	MnL_2Cl_2	Oct.,M-S	Antifungal	261
	Fe^{II}	FeL_3Cl_2	Oct.,$M-N(NH_2)$	"	261

METHODOLOGY

Chemicals

(1) Methanol

(2) Cobalt Chloride (CoOL2)

(3) Ether

(4) 2- aminothiazole

(5) Sodium bromide

(6) Sodium Iodide

Preparation of Complex:

1) 0.238m mole of (CocI2.6h2o) cobalt chloride hexa hydrted was dissolved in minimum amount of methanol. (.6 gm) mili moles of 2-amino thazole. ligand was dissolved in minimum amount of methanol. The mathematic solutions of the cobalt complex add into the solution of legend with continuous stirring. Heat the solution of water bath until the precipitate is appeared Dissolved this precipitate in ether (minimum amount). Now evaporate this Solution at water bath. Now fitter this Solution and obtained precipitate. Dried and weighed the precipitate.

Preparation of (CoL_2Br_2) Complex:

- A methenolic Solution of cobalt chloried hexa hydrated (.952 milimole) was added to the methenolic Solution of Sodium bromide (NaBr).(0.41156) milimole. Boil the Solution on wate bath untill the precipitate. of NacI obtained. Now filter thbè Solution. This Filterate was added in methenolic Solution on water bath for half an hour until precipitate, is obtained. Dissolved this precipitate. in small amount of methanol and filter it. Now dried and weighed.

Preparation of (CoL_2I_2 Complex):

- A methanolic Solution of Cobalt chloride hexohydratied (0.952 milimole) was added to the methenolix Solution of Sodium iodide (.599 milimole). Boiled this Solution on water bath' for half an hour until the precipitate. of Nal was obtained. Filter the Solution. Now this Filterate was added in methanolic solution of ligand (2 aminothiozole). (0.4 milimole). Now Boil this Solution on water bath. Until complex of (Col2l2) was obtained. Now Dissolved this precipitate. in minimum amount of methanol and filter it. Now Dried this precipitate and weighed.

SULFUR ESTIMATION

Chemicals :-

(1) Complex of CoL_2Cl_2 - 0.21 gm

(2) Complex of CoL_2Br_2 - 0.22 gm

(3) Complex of CoL_2I_2 - 0.25 gm

(4) Sodium Carbonate - 3 gm

(5) Sodium Nitrate - 2 gm

(6) Sodium Hydroxide - 1 gm

(7) Sodium Sulphate - (minimum amount)

(8) Dil HCL (for acidifying)

(9) Barium chloride (10%)

- Amount of Complex I (CoL_2CI_2-0.23 gm) and Sodium carbonate (3 gm), Sodium nitrate (2gm), sodium hydroxide added in a nickel crucible. Repeat this process with complex II (CoL_2Br_2 0.22 gm) and Complex Ill (CoL_2I_2 0.025 gm). Covered these Crucibles with the lid and heat on the Sand bath for 1 hr with slow heating. When these mixtures were melted. Completely than add sodium Sulphate to alkaline these mixtures. Put these nickel crucibles in 250 ml. beakers, and them added 150 ml water these beakers. Now boil these mixtures with constant starring. The melted mixture come out from the crucible into water. Now filter these solutions. Acidity with dil HCL. Then added 10% Barium chloride Solution in nickel crucibles (I, II, lII) until white precipitate of Barium Sulphate was obtained. Digested these solutions on. water bath. Cooled and filtered in a weighed G4 Crucibles. Dried these Crucibles in oven and weighed again.

RESULTAND DISCUSSION

CONDUCTIVITY

The molar conductivity value range from 20-50 ohm-1 om-2 mole-1 methonal shows that it is non electrolyte in the solvent.

S.No.	Complexes	% of Sulpher		Conductivity
		Theoretical	Experimental	
(1)	CoL_2Cl_2-$2H_2o$	18-333g	17.53g	$37.hm^{-1}Cm^{-2}$ $mole^{-1}$
(2)	CoL_2Br_2-$2H_2o$	17.67 gm	17.115 gm	$28.ohm^{-1}Cm^{-1}$ $mole^{-1}$
(3)	$Col_2l_2.4H_2o$	14.472 gm	14.035 gm	$45.ohm^{-1}Cm^{-2}$ $mole^{-1}$

International Journal of Research
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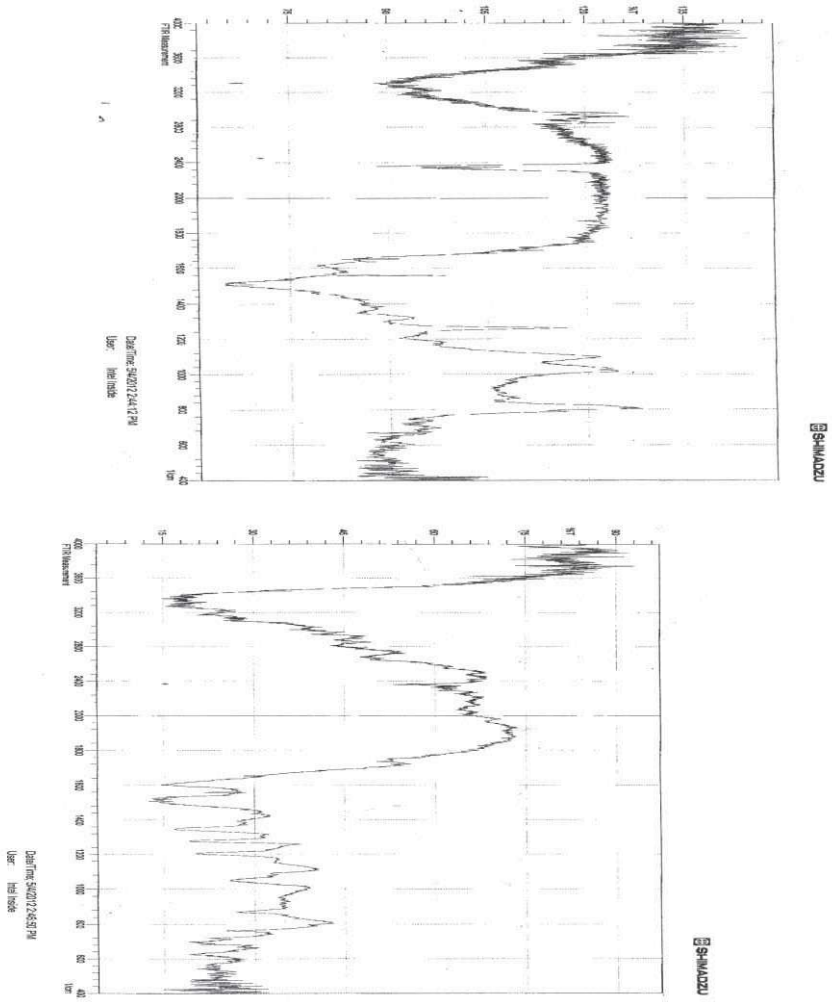
p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 08
July 2017



I.R. SPECTRA

2-Aminothazole has three potential doner sides. The exocyclic amino nitrogen, the ring nitrogen and a ring sulper atom. The ligand shows 2 IR bands at 3400 cm1 and 3290 cm1.Assigned to NH asymmetric and symmetric stretching mode respectively Coordination through amino - nitrogen is expected to show shift of the ligand NH stretching modes. It is observed that NH^- Symmtric and Asymmtric Stretching Bands remains almost unshfting in the Complex. The nature of the bands in the above region are not very clears which be due to the presence of some impurity in the complex.The NH_2 internal deformation mode generally occurs in the range (1650-1590 cm^{-1}) most workers have not commented on the change in the deformation mode as a diagnostic criteria for the bonding in the complexes of 2- aminohaterocyclic legends complexes of 2-aminohaterocyclic ligands. However a strong band at 1620 cm^{-1} was assigned to ΔNH_2 in 2- aminobenzothiazole (64)(9) can be a singned to (ΔNH_2). Which Shifts to shows no shifting in the complexes. However a shift of about 10^- in the ΔNH_2 frequencies is to be expected if the amino

group is involved in coordination. Therefore it is very likely that the amino nitrogen is not participation the bonding. The non indolent of amino group in

the bonding is also supported by the fact that NH^- Symmetric by the fact that ligand remain almost unshifted in the complex. It has been shown in the x-ray crystal study of Co (2-aminothiazole)$_2$ cl$_2$. That although Co-ordination in the compound occurs through the ring nitrogen there is extensive hydrogen bonding between the amino hydrogen and the Halogen atom bonded to the metal (Kc rapper) acts krist., B-37, 928, (1981).It appears that the C=N Stretching vibrations is generally couple with C=C vibration stretching and therefore it would be more scientific not to take off pure C=N Vibrations when such a configuration occurs with this limitation in mind one can say that the C=N Stretching vibrations in benzothiazole occurs at around (1560 cm^{-1}) (218) Crivsti atal (166) Attributed 1640 Cm^{-1} pand to C=N vibration. Mahapatra and Raman Rao a assigned the C=N Stretching Vibration of 2- aminobenzothiazole at around (1500 cm^{-1}) we assigned the band to the CH starching mode we observe a very strong band at (1515 cm^{-1}) xaminothiazole we shows a small decrees in the complexes as well as the nature of the band shows a split character. Therefore we tantatively assigned the linkage of the 2- amino thiazole with the metal via endocyclic nitrogen.

The C=H Starching mode is not, region indentify However the bands blw 800-700 cm^{-1} regain in 2- methy/benzothiazole (139), at (690 cm^{-1}) in 2-aminothiazole to be due to C=H stretching mode. (88-89) At we also observed bands b/w (655-610 cm^{-1}) in the ligand and assigned these band due to C=H Stretching mode. The intensity of the bond and position of the band does not show any change.

This may be due to the non involvement of sulpher in co-ordination. On comparing the ν NH^- asymmetric and symmetric bands ΔNH_2 ν C=N and ν C=s bands in the ligand of the complexes. The bonding through ring nitrogen of the ligand in the absence of others experimental data this assignment is purely tentative. As the spectra are recorded in 4000-400cm-1 we are not able to locate Co-N bands as these are expects below 400cm^{-1} region.

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e-ISSN: 2348-795X
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July 2017

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