

Preparation of co-ordination compounds of Pb^{II} with bis (2-pyrrolecarboxyaldehyde) cyclohexanediamine

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Abstract: This paper deals with preparation co-ordination compounds of Pb^{II} with bidentate ligand bis (2- pyrrolecarboxyaldehyde) cyclohexanediamine as MX₂L where M= Pb^{II}, X= Cl, Br, I and L= bis (2- pyrrole-carboxyaldehyde) cyclohexanediamine. The co-ordination compounds have been characterised on the basis of elemental analysis, conductivity, IR and x-ray photoelectron spectra. The structure of synthesized co-ordination compounds is trigonal bipyramidal.

(Keywords: Co-ordination Compounds, IR, Trigonal bipyramidal)

Introduction

The very easy interaction of lead from its ores made it one of the few metals used extensively from earliest times.¹

The most important one of lead in galena (PbS) which is widely distributed through the world.¹

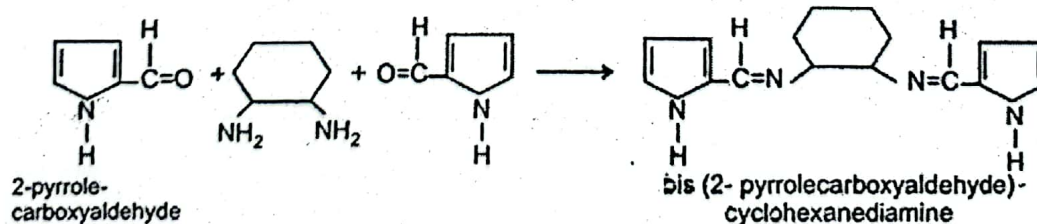
The literature survey of last three decades reveals that very little complexes are known of Lead (II) with Schiff-base ligands.^{2,3}

The bidentate ligand prepared by 2-pyrrolecarboxyaldehyde with cyclohexanediamine. This paper deals with synthesis of Pb(II) co-ordination compounds with bis (2- pyrrolecarboxyaldehyde) cyclohexanediamine.

Experimental

Preparation of bidentate ligand

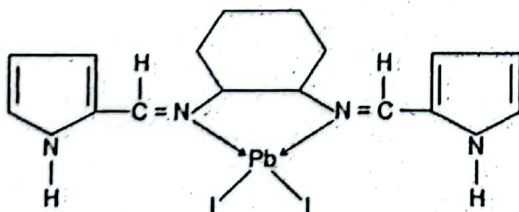
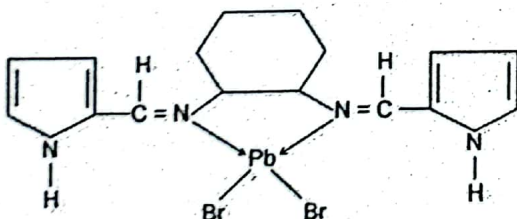
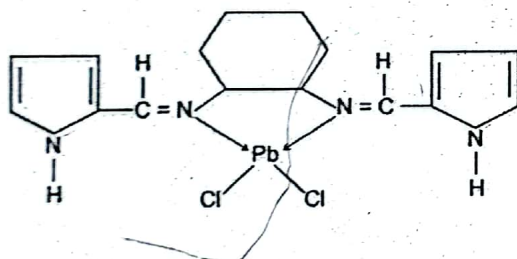
In 2mmol of 2- pyrrolecarboxyaldehyde 50 ml menthanol was mixed. 1mmol of cyclohexamediamine was refluxed for 3-4 hrs. The solid product was obtained, filtered, wased with menthanol Air-dried ligand were prepared as given in literature.⁴



Preparation of Co-ordination compounds as $PbX_2 \cdot L$:

In 1mmol of PbX_2 ($X = Cl, Br \text{ or } I$) 50 ml of $CHCl_3$ was mixed 1mmol of dry bis (2-pyrrolecarboxyaldehyde) cyclohexanediamine Schiff-base ligands was refluxed for 3-4 hrs. The solid product was obtained, filtered, washed with $CHCl_3$ and air dried.⁵

On the basis of physic-chemical studies of these compounds it can be concluded a structure as shown in Fig.



Results and Discussion

The newly synthesized Pb^{II} co-ordination compounds were light yellow solid and stable at room temp.

The elemental analysis were with in $\pm 0.5\%$ from C, H, N, X, Pb.

The analytical data of $PbX_2 \cdot L$ compounds.

S.N.	Compound	% found		
		C	H	N
1	$PbCl_2 \cdot L$	35	3.2	10.0
2	$PbBr_2 \cdot L$	30.2	2.6	8.6
3	$PbI_2 \cdot L$	26.2	2.4	7.6

The molar conductance data in DMF ($20-30 \text{ Ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$) of these compounds indicates that all these are non-ionic.⁶

All the prepared molecular adducts the ν_{C-N} modes shift to higher wave number from ligand ν_{C-N} $1610-1620 \text{ cm}^{-1}$ to molecular adduct ν_{C-N} $1635-1670 \text{ cm}^{-1}$, suggesting the co-ordination from N atom of the ligands.^{7,8}

Infrared frequency associated with fundamental modes of vibration of P_{b-N} and P_{b-Cl} was observed in the range of $305-330 \text{ cm}^{-1}$ and $280-285 \text{ cm}^{-1}$.⁶

The Pb2p and N1s binding energies (eV) data of PbX_2 and $PbX_2 \cdot L$ (where $X = Cl, Br, I$, $L = \text{bis (2-pyrrole-carboxyaldehyde) cyclohexanediamine}$) are listed in table.

Table : Pb2p, N1s binding energies (eV) in PbX₂ and PbX₂.L co-ordination compounds.

S.No.	Ligands, Salts, and Complexes	Pb2p	N1s Ligand
1	Ligand L		400.8
2	PbCl ₂	764.8	
3	PbCl ₂ .L	763.6	403.6
4	PbBr ₂	764.6	
5	PbBr ₂ .L	763.4	403.6
6	PbI ₂	764.4	
7	PbI ₂ .L	763.2	403.6

It is observed that the binding energies of Pb2p in starting material PbX₂ was higher than in the synthesized co-ordination compound [PbX₂.L]. These observation suggest that the electron density on Pb(II) metal ion increased due to co-ordination of ligand with Pb(II) metal ion.⁹

The value of N1s binding energy for N1s in co-ordination complexes is increased. This also concluded co-ordination of N atom with metal ion of PbX₂.¹⁰

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