

Synthesis and Characterization of $[(\text{PbX}_2)_2 \cdot \text{L}^1]$ and $[(\text{PbX}_2)_2 \cdot (\text{MCl}_2) \cdot \text{L}^1]$ Compounds with Macrocyclic Ligand

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We have synthesized different types of macrocyclic compounds $[(\text{PbX}_2)_2 \cdot \text{L}^1]$ and $[(\text{PbX}_2)_2 \cdot (\text{MCl}_2) \cdot \text{L}^1]$ with macrocyclic ligand (L^1) i.e. 36,9,12,20,23,26,29,35,36- decaazatricyclo 29.3.1.1¹⁴⁻¹⁸] hexatriaconta-1(34), 14,16,18(36) 31(35), 32-haxaene-2,13,19,30-tetraone ligand, where $\text{MCl}_2 = \text{CuCl}_2$ or CoCl_2 or MnCl_2 or NiCl_2 or ZnCl_2 . The all synthesized complexes were characterized by elemental analysis, molar conductance, IR and X-ray photoelectron spectra (XPS) data.

Keywords: Binuclear and Macrocyclic complexes, X-ray photoelectron spectroscopy, Heavy metals.

INTRODUCTION

The field of the macrocyclic chemistry of metals is developing very rapidly due to its application [1-3] and importance of macrocyclic metal complexes in coordination chemistry [4] and bioinorganic chemistry [5]. Many universal macrocyclic ligands such as crown ethers [6], porphyrines, saturated and unsaturated polyamines [7-9], polyazamacrocyclic [10,11], N_4S_2 donor macrocyclic [12,13], Robson type tetraaminodiphenol macrocyclic ligands [14,15] have been reported in past few decades. The studies on complexes of Schiff base macrocyclic ligands with different size, number and donor atoms for coordination with variety of metal centers have been studies [16]. The template condensation reaction lies in the core of macrocyclic chemistry [17]. Many transition metal ions in living systems work as enzymes and carrier in macrocyclic ligand environment [18] and used as modeling the active sites of metalloenzymes [19]. Macrocyclic metal complexes have been used as metal ion separation detecting tumor letions [20]; as in labeling monoclonal antibodies with radioactive models [21,22]; as cancer diagnosis [23-25]; as therapeutic and radio therapeutic [26]; as catalyst [27,28]; pharmacological agents [29,30]; as in biological process such as photosynthesis and dioxygen transport [31]; as photosensitizer [32]; as versatile coordination behaviour [33]; as toxicity against bacterial fungal

growth [34]; anticancerous [35]; antitumor [36]; as environmental importance as potential medicinal application such as contrast enhancing agents in magnetic resonance imaging (MRI) [37]; as NMR shift reagents and relaxation agents [38] and as RNA cleavage catalyst [39]. The design of host molecules are receptors for the recognition of substrates anion guest molecules in aqueous solution is very important target from an environment, industrial and health related points of view with multiple potential application [40]. The different types of macrocyclic ligands and their metal complexes discoveries have created supramolecular chemistry and its enormous diversity [41]. Recently few comprehensive reviews on macrocyclic ligands and their metal complexes with their application have been also appeared [42-46].

A literature survey reveals that many Pb(II) compound with monodentate, bidentate, tetradentate and Schiff base ligands have been synthesized and their structure have also been characterized by various physiochemical techniques [47]. But Pb(II) compounds with macrocyclic ligands are less known [48-50]. In this paper many Pb(II) compounds homobi lead(II) and heteromono lead(II)-monometal(II) compounds [where metal(II) = Cu(II), Co(II); Mn(II); Ni(II) and Zn(II)] with various macrocyclic ligands have been synthesized and characterized to stabilized their structure and geometry.

EXPERIMENTAL

Preparation of $[(\text{PbX}_2)_2\text{L}^1]$ compounds with macrocyclic ligands (L^1) i.e. 3,6,9,12,20,23,26,29,35,36-decaazatricyclo 29.3.1.1¹⁴⁻¹⁸] hexatriaconta-1(34),14,16,18(36),31(35),32-haxaene-2,13,19,30-tetraone ligands: Triethylenetetraamine (1 mmol) in dry ethanol and $\text{PbCl}_2/\text{PbBr}_2$ (1 mmol) was refluxed for 1 h and then put drop-wise pyridine 2,6-dicarboxylic acid into this solution (2 mmol:2 mmol) ratio and again refluxed this solution for 2 h. The resulting precipitate was filtered and recrystallized into ether:ethanol (9:1 ratio) (Fig. 1).

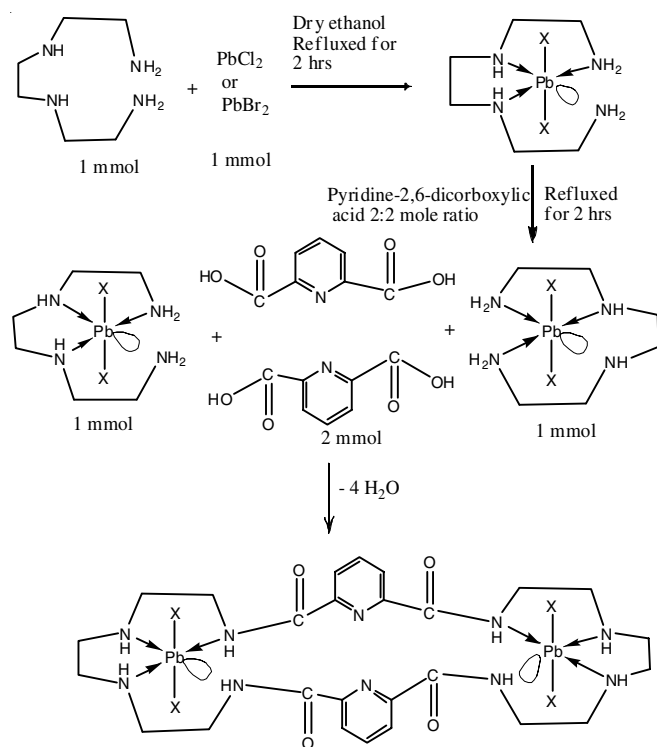


Fig. 1. Proposed mechanism for homobimetallic compound $[(\text{PbX}_2)_2\text{L}^1]$

Preparation of $[(\text{PbX}_2)(\text{MCl}_2)\text{L}^1]$ (where $\text{M} = \text{Cu(II)}$, Co(II) , Mn(II) , Ni(II) and Zn(II)) compounds with macrocyclic ligands L^1 i.e. 3,6,9,12,20,23,26,29,35,36-decaazatricyclo[29.3.1.1¹⁴⁻¹⁸] hexatriaconta-1(34),14,16,18(36),31(35),32-hexaene-2,13,19,30-tetraamine ligands: Triethylenetetraamine (1 mmol) was mixed with PbCl_2 or PbBr_2 (1 mmol) in dry ethanol and refluxed for 1 h (solution A). Again triethylenetetraamine (1 mmol) was mixed with MCl_2 (where $\text{MCl}_2 = \text{CuCl}_2$ or CoCl_2 or MnCl_2 or NiCl_2 or ZnCl_2) (1 mmol) in dry ethanol and refluxed for 1 h (solution B). In solution A then put drop-wise pyridine dicarboxylic acid (2 mmol) and refluxed for 1 h and then put drop-wise solution B into it (1 mmol) and refluxed again for 2 h. The resulting precipitate was filtered and recrystallized into ether:ethanol (9:1 ratio) (Fig. 2).

RESULTS AND DISCUSSION

All these newly synthesized compounds $[\text{PbX}_2\text{L}^1]$ and $[\text{PbX}_2(\text{MCl}_2)\text{L}^1]$ (where $\text{M} = \text{Cu(II)}$, Co(II) , Mn(II) , Ni(II) and Zn(II)) are light pink colour solids. They are soluble in

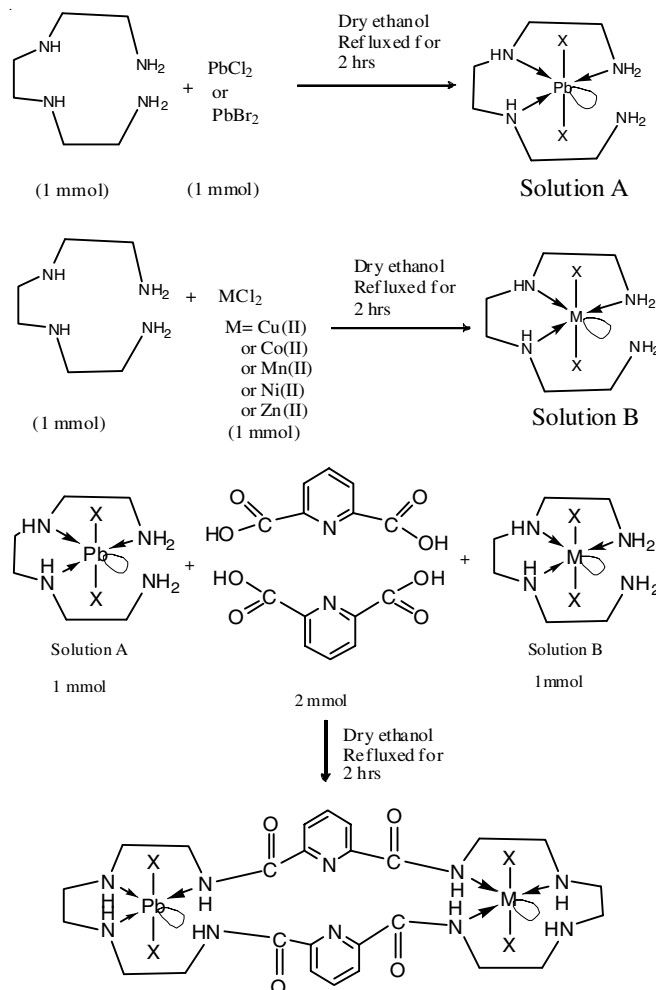


Fig. 2. Proposed mechanism for heterobimetallic compound $[(\text{PbX}_2)(\text{MX}_2)\text{L}^2]$

common organic solvents and stable towards atmospheric oxygen and moisture. They all are stable at room temperature even on long standing. The elemental analysis for C, H, N were found within $\pm 0.5\%$ and molar conductance values of compounds in 10^{-3}M were observed in range from $20\text{--}30\text{ ohm}^{-1}\text{cm}^2\text{mol}^{-1}$ in acetone and $0.82\text{ to }1.02\text{ ohm}^{-1}\text{cm}^2\text{mol}^{-1}$ in nitrobenzene indicating absence of ionic species [51] (Table-1). Infrared frequency associated with fundamental modes of vibration of $\nu(\text{Pb-N})$ and $\nu(\text{Pb-Cl})$ was observed in the range $330\text{--}305\text{ cm}^{-1}$ and $280\text{--}285\text{ cm}^{-1}$, respectively, in these all prepared compounds [51]. $\nu(\text{Cu-Cl})$ in $[\text{PbX}_2(\text{CuCl}_2)\text{L}^1]$; $\nu(\text{Co-Cl})$ in $[(\text{PbX}_2(\text{CoCl}_2))\text{L}^1]$; $\nu(\text{Mn-Cl})$ in $[\text{PbX}_2(\text{MnCl}_2)\text{L}^1]$; $\nu(\text{Ni-Cl})$ in $[\text{PbX}_2(\text{NiCl}_2)\text{L}^1]$ and $\nu(\text{Zn-Cl})$ in $[\text{PbCl}_2(\text{ZnCl}_2)\text{L}^1]$ compounds were observed in ranges $285\text{--}280\text{ cm}^{-1}$ [41].

The binding energies (eV) of PbX_2 ; $[\text{PbX}_2\text{L}^1]$; $[(\text{PbX}_2)(\text{MCl}_2)\text{L}^1]$ (where $\text{M} = \text{Cu(II)}$, Co(II) , Mn(II) , Ni(II) and Zn(II)); for $\text{Pb } 3p_{1/2,3/2}$ $\text{M } np$ ($\text{M} = \text{metal}$) and $\text{N } 1s$ photoelectron peaks are listed in Tables 2 and 3 (Figs. 3-6). It was observed that the binding energies of $\text{Pb } 3p_{1/2,3/2}$ in starting material PbCl_2 or PbBr_2 was higher than their prepared molecular adducts i.e. $[(\text{PbX}_2)\text{L}^1]$ and $[(\text{PbX}_2)(\text{MCl}_2)\text{L}^1]$. These observation suggested that electron density on the Pb(II) metal ion has increased due to coordination of ligands with Pb(II) metal ion [52] (Figs. 3 and 4). It was also observed that binding energies of $\text{M } 2p_{1/2,3/2}$ in MCl_2 was higher than their prepared molecular adducts of

TABLE-1
ANALYTICAL DATA OF $[(\text{PbX}_2)_2.\text{L}^1]$ AND $[(\text{PbX}_2)(\text{MX}_2).\text{L}^1]$ COMPOUNDS

S. No.	Compound	Elemental analysis (%): Found (calcd.)				Molar conductance in acetone ($\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$)
		Pb	C	H	N	
1	$[(\text{PbCl}_2).\text{L}^1]$	37.6 (37.7)	27.2 (27.3)	3.4 (3.4)	12.6 (12.7)	20
2	$[(\text{PbCl}_2)(\text{CuCl}_2).\text{L}^1]$	20.0 (20.2)	29.4 (29.3)	3.6 (3.7)	13.4 (13.6)	24
3	$[(\text{PbCl}_2)(\text{CoCl}_2).\text{L}^1]$	21.6 (21.7)	31.4 (31.5)	4.0 (4.0)	14.6 (14.7)	28
4	$[(\text{PbCl}_2)(\text{MnCl}_2).\text{L}^1]$	21.8 (21.8)	31.6 (31.7)	4.0 (4.0)	14.8 (14.8)	26
5	$[(\text{PbCl}_2)(\text{NiCl}_2).\text{L}^1]$	21.6 (21.8)	31.4 (31.6)	4.0 (4.0)	14.6 (14.7)	24
6	$[(\text{PbCl}_2)(\text{ZnCl}_2).\text{L}^1]$	21.6 (21.8)	32.2 (31.3)	3.8 (3.9)	14.4 (14.6)	30
7	$[(\text{PbBr}_2).\text{L}^1]$	21.6 (21.6)	23.4 (23.5)	2.8 (2.9)	10.8 (10.9)	26
8	$[(\text{PbBr}_2)(\text{CuCl}_2).\text{L}^1]$	32.2 (32.4)	28.6 (28.7)	3.6 (3.6)	13.2 (13.4)	24
9	$[(\text{PbBr}_2)(\text{CoCl}_2).\text{L}^1]$	19.6 (19.8)	28.6 (28.8)	3.6 (3.6)	13.4 (13.4)	22
10	$[(\text{PbBr}_2)(\text{MnCl}_2).\text{L}^1]$	20.0 (20.0)	28.8 (28.9)	3.6 (3.6)	13.4 (13.5)	20
11	$[(\text{PbBr}_2)(\text{NiCl}_2).\text{L}^1]$	19.8 (19.8)	28.8 (28.8)	3.6 (3.6)	13.4 (13.4)	24
12	$[(\text{PbBr}_2)(\text{ZnCl}_2).\text{L}^1]$	19.6 (19.8)	28.6 (28.6)	3.6 (3.6)	13.2 (13.3)	26

TABLE-2
Pb $3p_{1/2,3/2}$ BINDING ENERGIES (eV) IN PbX_2 ($\text{X} = \text{Cl}, \text{Br}$) AND $[(\text{PbX}_2).\text{L}^1]$; $[(\text{PbX}_2)(\text{MCl}_2).\text{L}^1]$ COMPOUNDS

S. No.	Compound	Pb $3p_{1/2,3/2}$		$\text{M} = \text{P}_{1/2,3/2}$	
		Pb $3p_{1/2}$	Pb $3p_{3/2}$		
1	PbCl_2	560.8	532.8	—	—
2	PbBr_2	560.6	532.6	—	—
3	$[(\text{PbCl}_2).\text{L}^1]$	559.8	531.8	—	—
4	$[(\text{PbBr}_2).\text{L}^1]$	559.6	531.6	—	—
5	CuCl_2	—	—	952.8	932.8
6	$[(\text{PbCl}_2)(\text{CuCl}_2).\text{L}^1]$	559.8	531.8	951.6	931.6
7	$[(\text{PbCl}_2)(\text{CuCl}_2).\text{L}^1]$	559.6	531.8	951.6	931.6
8	CoCl_2	—	—	795.8	781.8
9	$[(\text{PbCl}_2)(\text{CoCl}_2).\text{L}^1]$	559.8	531.8	794.8	780.8
10	$[(\text{PbBr}_2)(\text{CoCl}_2).\text{L}^1]$	559.6	531.6	794.8	780.8
11	MnCl_2	—	—	653.8	642.8
12	$[(\text{PbCl}_2)(\text{MnCl}_2).\text{L}^1]$	559.8	531.8	652.8	641.8
13	$[(\text{PbBr}_2)(\text{MnCl}_2).\text{L}^1]$	559.6	531.6	652.6	641.6
14	NiCl_2	—	—	873.8	856.8
15	$[(\text{PbCl}_2)(\text{NiCl}_2).\text{L}^1]$	559.8	531.8	872.8	855.8
16	$[(\text{PbBr}_2)(\text{NiCl}_2).\text{L}^1]$	559.6	531.6	872.8	855.8
17	ZnCl_2	—	—	1045.8	1022.8
18	$[(\text{PbCl}_2)(\text{ZnCl}_2).\text{L}^1]$	559.8	531.8	1044.8	1021.8
19	$[(\text{PbBr}_2)(\text{ZnCl}_2).\text{L}^1]$	559.6	531.6	1044.8	1021.8

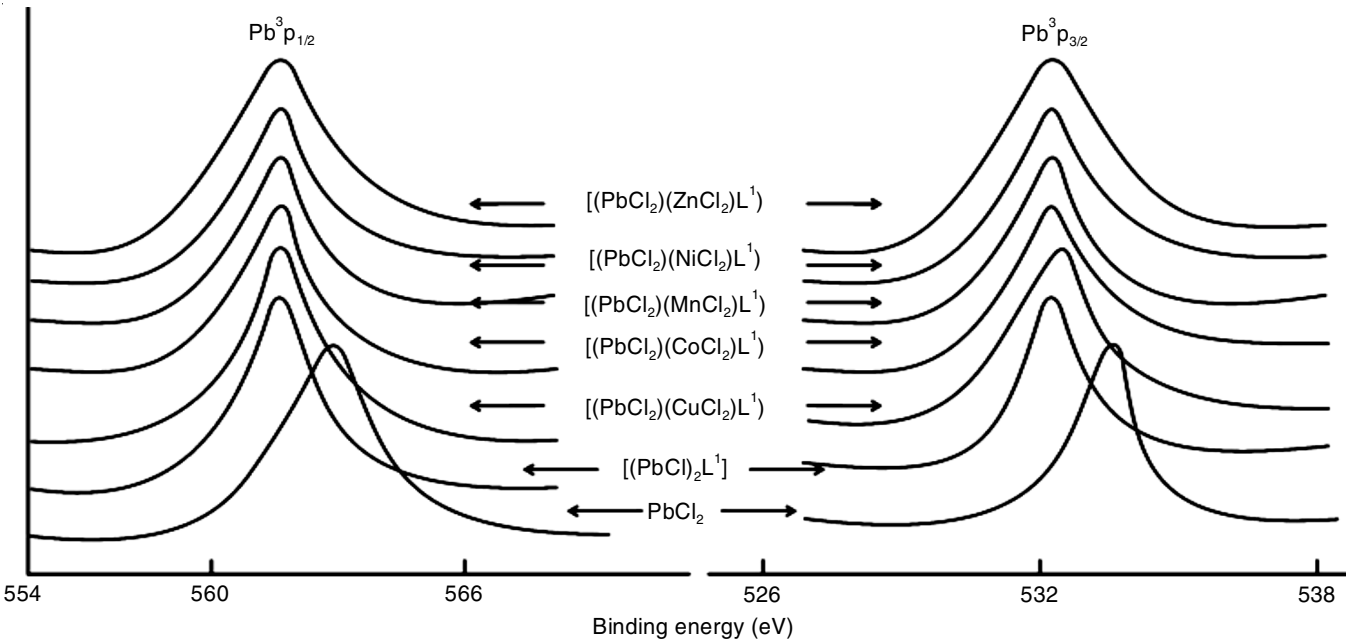


Fig. 3. Pb $3p_{1/2,3/2}$ binding energies (eV) in PbX_2 ($\text{X} = \text{Cl}, \text{Br}$) and $[(\text{PbX}_2).\text{L}^1]$; $[(\text{PbX}_2)(\text{MCl}_2).\text{L}^1]$ compounds

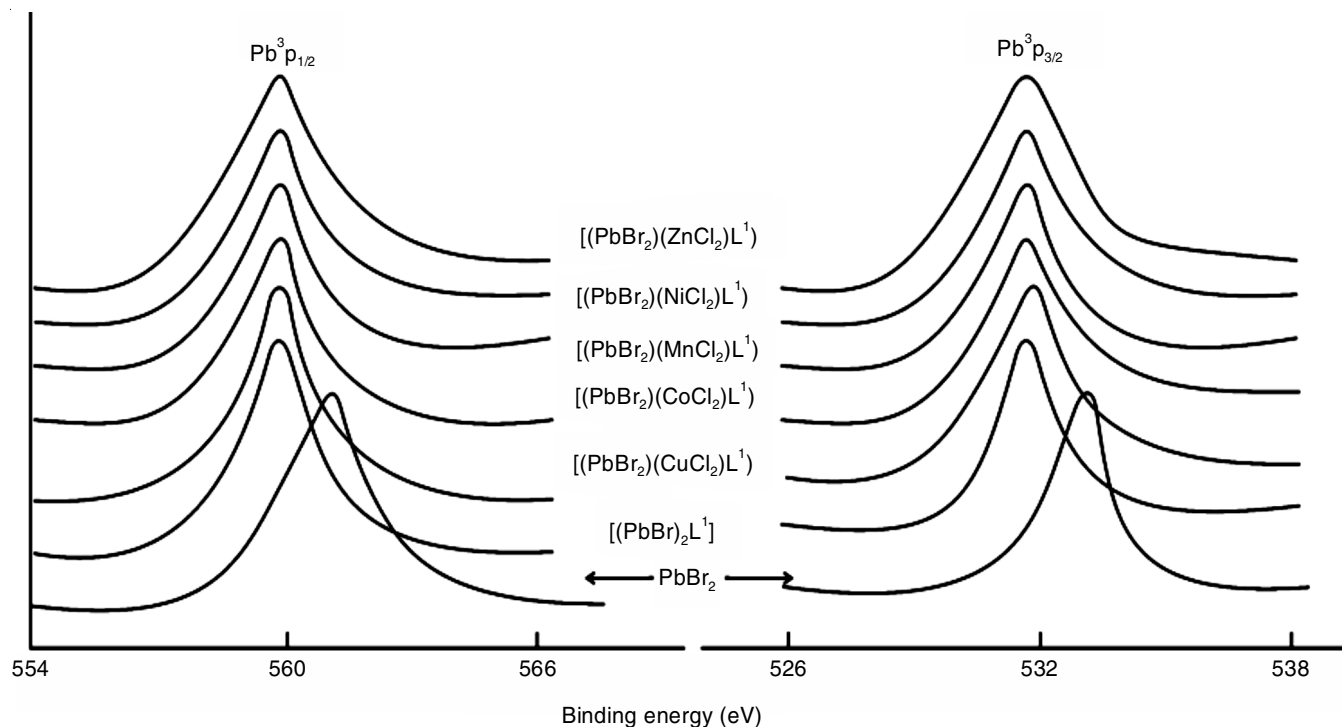
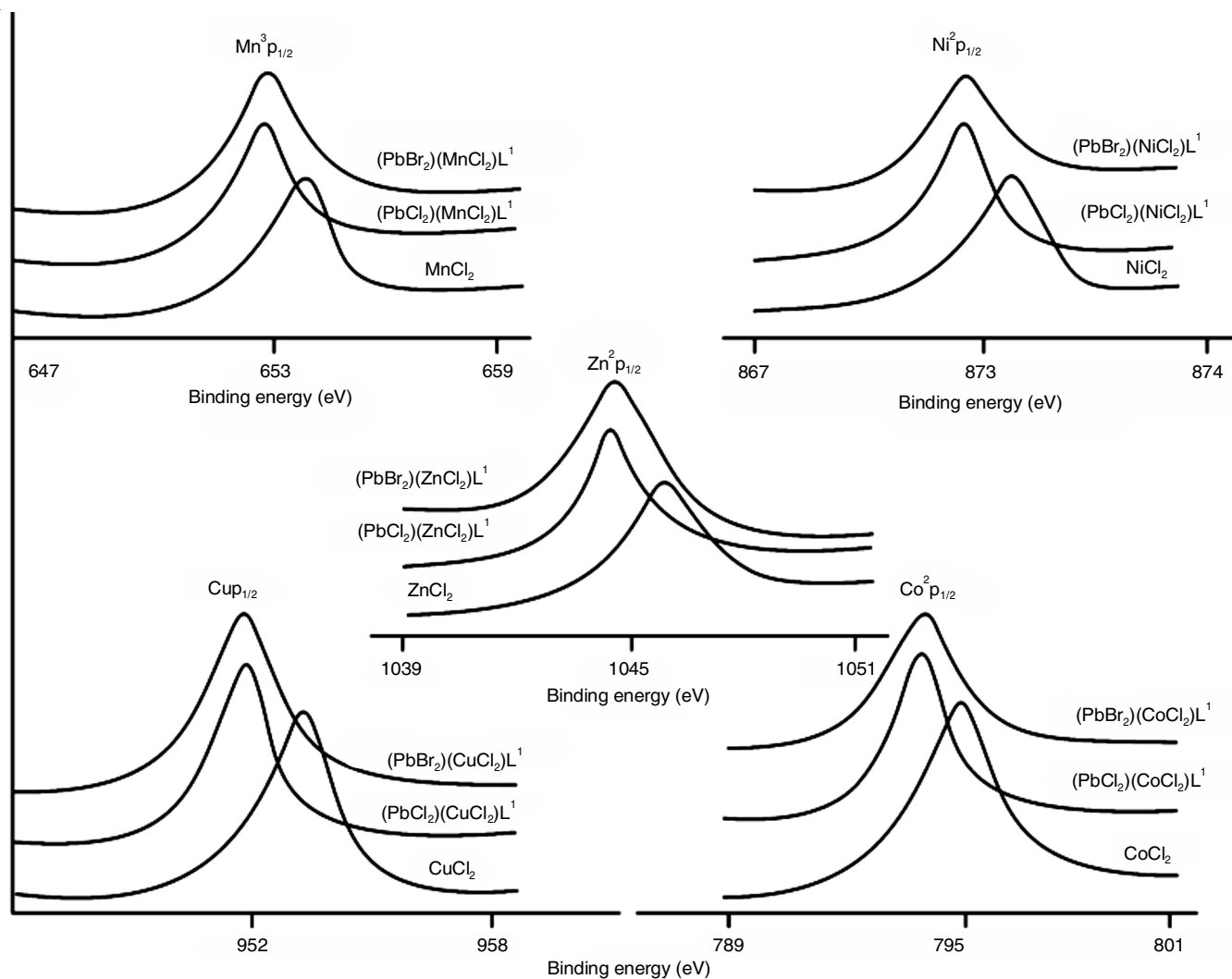
Fig. 4. N 1s binding energies (eV) in $[(\text{PbX}_2)_2\text{L}^1]$ and $[(\text{PbX}_2)_2(\text{MX}_2)\text{L}^1]$ compoundsFig. 5. M 2p_{1/2} binding energies (eV) in $[(\text{PbX}_2)(\text{MCl}_2\text{L}^1)]$ compounds

TABLE-3
N1s BINDING ENERGIES (eV) IN $[(\text{PbX}_2)_2\text{L}^1]$
AND $[(\text{PbX}_2)_2(\text{MX}_2)\text{L}^1]$ COMPOUNDS

S. No.	Compounds	N1s	
		Uncoordinated	Coordinated
1	$[(\text{PbCl}_2)_2\text{L}^1]$	399.6(4)	403.4(6)
2	$[(\text{PbCl}_2)(\text{CuCl}_2)\text{L}^1]$	399.6(3)	403.4(7)
3	$[(\text{PbCl}_2)(\text{CoCl}_2)\text{L}^1]$	399.6(3)	403.4(7)
4	$[(\text{PbCl}_2)(\text{MnCl}_2)\text{L}^1]$	399.6(3)	403.4(7)
5	$[(\text{PbCl}_2)(\text{NiCl}_2)\text{L}^1]$	399.6(3)	403.4(7)
6	$[(\text{PbCl}_2)(\text{ZnCl}_2)\text{L}^1]$	399.6(3)	403.4(7)
7	$[(\text{PbBr}_2)_2\text{L}^1]$	399.6(4)	403.4(6)
8	$[(\text{PbBr}_2)(\text{CuCl}_2)\text{L}^1]$	399.6(3)	403.4(6)
9	$[(\text{PbBr}_2)(\text{CoCl}_2)\text{L}^1]$	399.6(3)	403.4(6)
10	$[(\text{PbBr}_2)(\text{MnCl}_2)\text{L}^1]$	399.6(3)	403.4(6)
11	$[(\text{PbBr}_2)(\text{NiCl}_2)\text{L}^1]$	399.6(3)	403.4(6)
12	$[(\text{PbBr}_2)(\text{ZnCl}_2)\text{L}^1]$	399.6(3)	403.4(6)

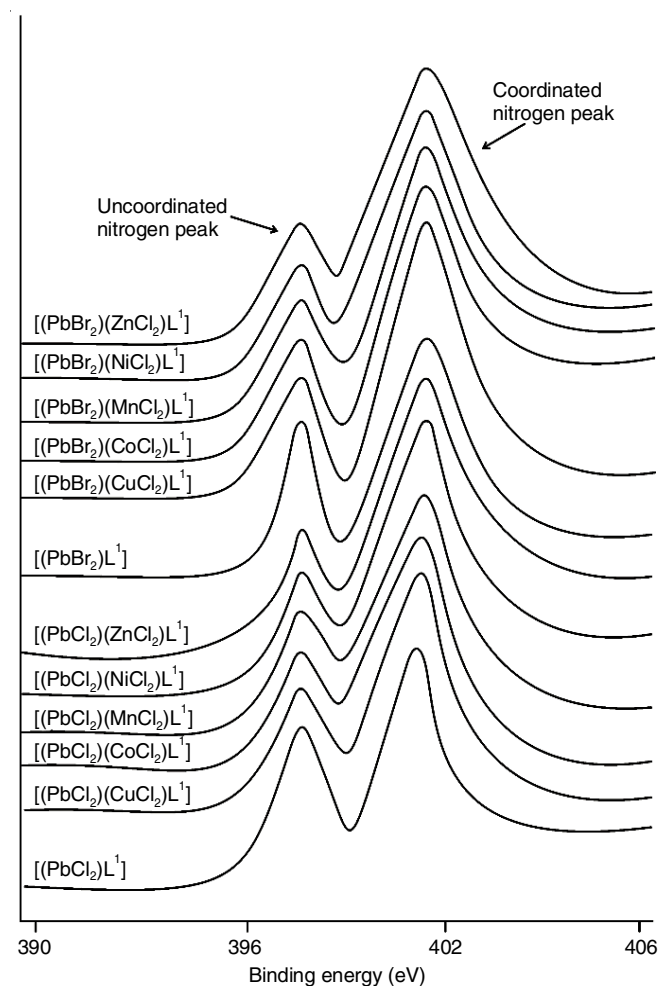


Fig. 6. N 1s binding energies (eV) in $[(\text{PbX}_2)_2\text{L}^1]$ and $[(\text{PbX}_2)_2(\text{MX}_2)\text{L}^1]$ compounds

the type $[(\text{PbX}_2)(\text{MX}_2)\text{L}^1]$; suggested that electron density on metal ion $\text{M} = \text{Cu(II)}, \text{Co(II)}, \text{Ni(II)}, \text{Mn(II)}$ and Zn(II) has increased due to coordination of ligands with these metal ions [52] (Fig. 5). More over, N1s photoelectron peaks with intensity ratio 4:6 in $[(\text{PbX}_2)_2\text{L}^1]$; 3:7 in $[(\text{PbX}_2)(\text{MCl}_2)\text{L}^1]$ at 399.6 eV and at 403.4 eV suggested in $[(\text{PbX}_2)_2\text{L}^1]$ four nitrogen atoms are uncoordinated and six are coordinated; in $[(\text{PbX}_2)(\text{MCl}_2)\text{L}^1]$ three nitrogen are uncoordinated and seven nitrogen are coordinated [52] (Fig. 6) (Table-3).

On the basis of elemental analysis, molar conductance data showing that all these molecular adducts are nonionic and IR and X-ray photoelectron spectra (XPS) results, showing the site of coordination, structure of these prepared molecular adducts are assigned and an octahedral geometry are established.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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