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Formation and description of the coordination compounds of N-(2-hydroxymethylphenyl)-C-(3'-carboxy-2'-hydroxyphenyl) thiazolidin-4-one

Dr. Sharad Kumar Jha

Abstract

A dry benzene solution of the Schiff base N-(2-hydroxymethylphenyl)-3'-carboxy-2'-hydroxybenzylideneimine upon reacting with mercaptoacetic acid undergoes cyclization and forms N-(2-hydroxymethylphenyl)-C-(3'-carboxy-2'-hydroxyphenyl) thiazolidin-4-one, LH₃ (I). A MeOH solution of I reacts with Co(II), Ni(II), Cd(II), Zr(OH)₂(IV), and UO₂(VI) ions and forms the monomeric coordination compounds, [M(LH)(MeOH)₃] [where M = Co(II), Ni(II)], [M'(LH)(MeOH)] [here M' = Cd(II), UO₂(VI)] and [Zr(OH)₂(LH)(MeOH)]. The coordination compounds have been characterized on the bases of elemental analyses, molar conductance, molecular weight, spectral (IR, NMR, and reflectance) studies, and magnetic susceptibility measurements. I behaves as a dibasic tridentate OOS donor ligand in these compounds. The compounds are nonelectrolytes ($A_M = 3.8-8.9$ mho cm² mol⁻¹) in DMF.

Keywords: Parental attitude, participation, sports, girls

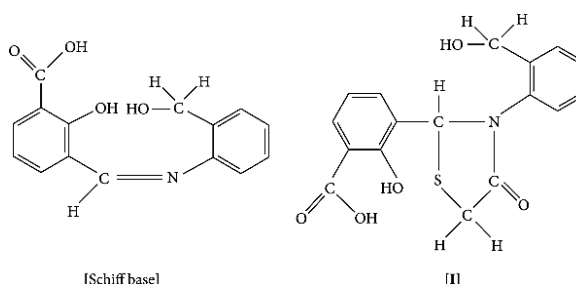
1. Introduction

The chemistry of thiazolidin-4-ones has been studied extensively due to their versatile biological properties [1]. Thiazolidin-4-ones, a saturated form of thiazole with carbonyl group on fourth carbon [2], have biological activities like those that are antipsychotic [3], antitubercular [4], antibacterial [5], anticonvulsant [6], antifungal [7], amoebicidal [8], antioxidant [9], antibiotic [10], and so forth.

Literature survey shows that much of them has been reported on the syntheses and [11] of a variety of thiazolidin-4-ones, but very less is known about their coordination compounds [12-14]. Metal complexes play an important role in plant and animal life due to their physicochemical and biological properties. Metal ions are involved in specific interactions with antibiotics, proteins, nucleic acids, and other biomolecules [15]. Most of the drugs have improved pharmacological properties in the form of their metal complexes. Transition metal ions play a very important role in the pharmacological action of metal-based drugs and these drugs are more effective against infectious microbes than the uncomplexed drugs [16].

These facts motivate us to explore the coordination behavior of a newly synthesized thiazolidin-4-one with some transition metal ions.

In this paper, we describe the of N-(2-hydroxymethylphenyl)-C-(3'-carboxy-2'-hydroxyphenyl)thiazolidin-4-one, LH₃ (I) and its coordination compounds with Co(II), Ni(II), Cd(II), Zr(OH)₂(IV), and UO₂(VI) ions. The structure of Schiff base and thiazolidin-4-one, LH₃ (I) is shown in Figure 1.



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2. Experimental

2.1. Materials. *o*-Aminobenzylalcohol [Aldrich]; nickel(II) acetate tetrahydrate, cadmium(II) acetate dihydrate, dioxouranium (VI) acetate tetrahydrate (Sarabhai); cobalt(II) acetate tetrahydrate, hexadecaquaoctahydroxotetrazirconium (IV) chloride (BDH); methanol, ethanol, mercaptoacetic acid, dry benzene, and sodium bicarbonate (Ranbaxy) were used as supplied for the syntheses. 3-Formylsalicylic acid [17] and hexadecaquaoctahydroxotetrazirconium (IV) acetate [18] were synthesized by the following reported procedures.

2.3. Synthesis of the *N*-(2-hydroxymethylphenyl)-3'-carboxy-2'-hydroxybenzylidene-neimine (Schiff Base). A MeOH solution (30 mL) of *o*-aminobenzylalcohol (1.23 g, 10 mmol) was added to a MeOH solution (30 mL) of 3-formylsalicylic acid (1.66 g, 10 mmol) and the mixture was then refluxed for 1 h. The precipitates formed were suction filtered, washed with MeOH, and dried in *vacuo* at room temperature over silica gel for 24 h. Yield = 58%. The elemental analyses of the compound gave the satisfactory results.

2.4. Synthesis of *N*-(2-hydroxymethylphenyl)-C-(3'-carboxy-2'-hydroxyphenyl)thiazolidin-4-one (I). A dry benzene solution of the Schiff base (2.71 g, 10 mmol) and

mercaptoacetic acid (0.92 g, 10 mmol) were refluxed for 12h in a water bath. The mixture was cooled to room temperature and then was washed with 10% NaHCO₃ solution. The benzene layer was separated by using a separating funnel. The partial evaporation of the benzene layer gave a solid product, which was filtered, washed with petroleum ether and recrystallized from petroleum ether. The compounds were dried as mentioned above. Yield = 14%. Anal: (I, C₁₇H₁₅NO₅S) (obsd: C, 58.91%; H, 4.37%; N, 4.12%; S, 9.11%. calc.: C, 59.13%; H, 4.35%; N, 4.06%; S, 9.28%); IR bands (KBr): 2860 cm⁻¹ [ν(O-H)(intramolecular H-bonding)], 1710 cm⁻¹ [ν(C=O)(thiazolidinone ring)], 1675 cm⁻¹ [ν(C=O)(carboxylic)], 1570 cm⁻¹ [ν(C-N)(thiazolidinone ring)], 1520 cm⁻¹ [ν(C-O)(phenolic)], 1225 cm⁻¹ [ν(C-O)(alcoholic)], and 830 cm⁻¹ [ν(C-S)(thiazolidinone ring)].

2.5. Syntheses of Coordination Compounds of I. A MeOH solution (30-50 mL) of the appropriate metal salt (10 mmol) was added to a MeOH solution (50 mL) of I (3.45 g, 10 mmol) and the mixture was then refluxed for 3-4 h. The solid products formed were suction filtered, washed with MeOH and recrystallized from MeOH and were then dried as mentioned above. Yield = 50-75%.

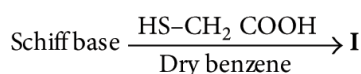
Table 1:0 Analytical, molar conductans (Δ_M), and molecular weight data of I and its coordination compounds

Compound	Mol. formula	Δ (mho cm ² Mol ⁻¹)	Mol. wt obsd (calcd)	Obsd (calcd)9%				
				M	C	H	N	S
LH ₃ (I)	C ₁₇ H ₁₅ NO ₅ S	-	345 ^a (345.0)	-	58.91 (59.13)	4.37 (4.35)	4.12 (4.06)	9.11 (9.28)
[Co(LH)(MeOH) ₃]	CoC ₂₀ H ₂₃ NO ₈ S	4.8	511.6 ^b (497.9)	11.64 (11.83)	48.37 (48.20)	5.10 (5.02)	2.74 (2.81)	6.32 (6.43)
[Ni(LH)(MeOH) ₃]	NiC ₂₀ H ₂₃ NO ₈ S	3.8	481.3 ^b (497.7)	11.56 (11.79)	48.17 (48.22)	5.17 (5.02)	2.72 (2.81)	6.28 (6.43)
[Cd(LH)(MeOH)]	CdC ₁₈ H ₁₇ NO ₆ S	7.7	511.2 ^b (487.4)	23.17 (23.06)	44.50 (44.32)	3.53 (3.49)	2.72 (2.87)	6.49 (6.56)
[Zr(OH) ₂ (LH)(MeOH)]	ZrC ₁₈ H ₁₉ NO ₈ S	8.9	517.2 ^b (500.2)	18.06 (18.23)	43.34 (43.18)	3.74 (3.80)	2.85 (2.80)	6.32 (6.40)
[UO ₂ (LH)(MeOH)]	UC ₁₈ H ₁₇ NO ₈ S	6.8	659.5 ^b (645.0)	36.67 (36.90)	33.24 (33.49)	2.45 (2.64)	2.05 (2.17)	4.85 (4.96)

^aMass spectral data. ^bRast method data.

Table 2: IR, reflectance spectral data (cm⁻¹), and magnetic moments of the coordination compounds of I.

Compound	ν _{as} (COO)	ν _s (COO)	ν(C-O) (phenolic)	ν(C-S)	ν(C-O) (MeOH)	ν _{max}	Mag. moment (B.M)
LH ₃ (I)	-	-	1520	830	-	-	Diamagnetic
[Co(LH)(MeOH) ₃]	1572	1358	1530	795	986	8700,13000,18550	4.75
[Ni(LH)(MeOH) ₃]	1572	1352	1526	805	989	8980,15980,24900	3.14
[Cd(LH)(MeOH)]	1560	1347	1530	810	984	-	Diamagnetic
[Zr(OH) ₂ (LH)(MeOH)]	1562	1337	1530	815	974	-	Diamagnetic
[UO ₂ (LH)(MeOH)]	1567	1349	1529	810	980	-	Diamagnetic



Scheme 1: Preparative scheme of LH₃(I).

3. Results and Discussion

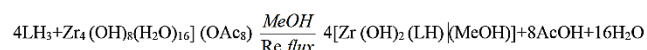
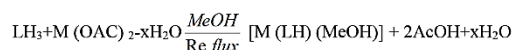
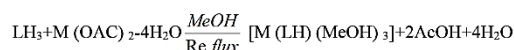
A dry benzene solution of the Schiff base reacts with mercaptoacetic acid and forms *N*-(2-hydroxymethylphenyl)-C-(3'-carboxy-2'-hydroxyphenyl)thiazolidin-4-one, LH₃ (I). The reaction of I with appropriate metal salt in 1 : 1 molar ratio in MeOH produces the coordination compounds, [M(LH)(MeOH)₃] [where M = Co(II)], [M'(LH)(MeOH)] [where M' = Cd(II)], and [Zr(OH)₂(LH)(MeOH)]. The formations of I from the Schiff base and the coordination compounds of I take place according to Schemes 1 and 2.

The coordination compounds are air-stable at room temperature. They are insoluble Highly polar in H₂O, partially soluble in slightly polar molecules MeOH, EtOH, and completely soluble in DMSO and DMF. Their molar

conductance measurements (Λ_M = 3.8-8.9 mho cm² mol⁻¹) in DMF indicate their nonelectrolytic nature. The analytical data of I and its coordination compounds are presented in Table 1.

3.1. Infrared Spectral Studies. The infrared spectra of I and its coordination compounds were recorded in KBr and the prominent peaks (in cm⁻¹) are shown in Table 2. The Schiff base exhibits the ν(C=N) (azomethine) stretch at 1640 cm⁻¹. This band disappears in I and a new band appears at 1570 cm⁻¹ due to the ν(C-N)(thiazolidinone ring) stretch [19] indicating the conversion of the Schiff base into I. The formation of I is further supported by the appearance of a new band at 830 cm⁻¹ due to the ν(C-S) (thiazolidinone ring) stretch [20]. A negative shift of 15-35 cm⁻¹ of the ν(C-S) (thiazolidinone ring) stretch in the coordination compounds indicates the involvement of the S atom of the thiazolidinone moiety in coordination [14]. I shows the ν(C=O) (thiazolidinone ring) stretch [20] at 1710 cm⁻¹. This band remains unchanged in the coordination compounds

indicating the noninvolvement of O atom in the coordination. The $\nu(\text{C-O})$ (alcoholic) stretch [21] of I occurs at 1225 cm^{-1} which remains unchanged in the complexes. I exhibits a strong band at 2860 cm^{-1} due to the intramolecular H-bonded OH group of phenolic and/or carboxylic acid moieties [22]. This band disappears in the coordination compounds indicating the breakdown of H-bonding and subsequent deprotonation of the OH group followed by the involvement of phenolic and carboxylic acid O atoms in coordination. The presence of a broadband at $\sim 3400\text{ cm}^{-1}$ due to $\nu(\text{O-H})$ (MeOH) and the decrease of $\nu(\text{C-O})$ (MeOH) stretch from 1034 cm^{-1} to lower energy by $45\text{--}60\text{ cm}^{-1}$ in the coordination compounds of I indicate the



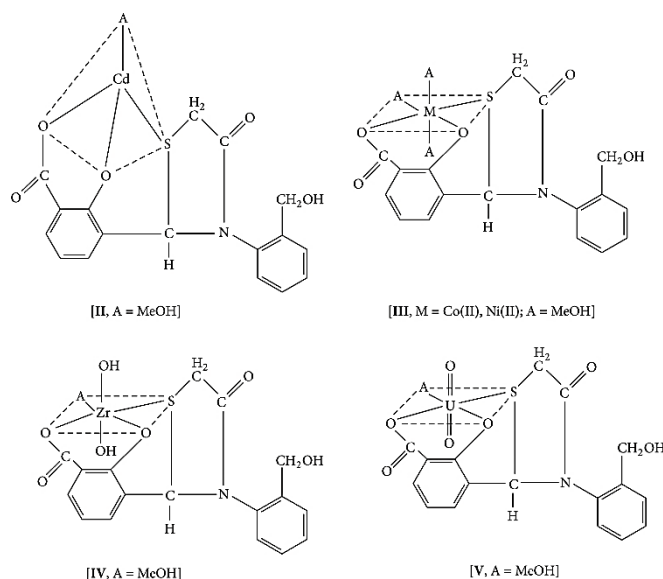
Scheme 2: Preparative scheme of coordination compounds of LH_3 (I).

involvement of the O atom of MeOH in coordination [23]. The appearance of two new bands between $1560\text{--}1572\text{ cm}^{-1}$, $\nu_{\text{as}}(\text{COO})$ and $1337\text{--}1358\text{ cm}^{-1}$, $\nu_{\text{s}}(\text{COO})$ stretches indicates the presence of the coordinated carboxylate group in the coordination compounds. The energy difference ($\Delta\nu = 213\text{--}225\text{ cm}^{-1}$) between these stretches is $>210\text{ cm}^{-1}$ which indicates the monodentate nature of the carboxylate moiety [24]. The $\nu(\text{C-O})$ stretch of I occurs at 1520 cm^{-1} . This band shifts to higher energy by $6\text{--}10\text{ cm}^{-1}$ in the coordination compounds indicating the involvement of phenolic O atom of 3-formylsalicylic acid moiety in coordination [23]. The absence of a band between $835\text{--}955\text{ cm}^{-1}$ due to the $\nu(\text{Zr=O})$ stretch [25] in the present $\text{Zr}(\text{OH})_2(\text{IV})$ compound suggests its formulation as $[\text{Zr}(\text{OH})_2(\text{LH})(\text{MeOH})]$ and not as $[\text{Zr}(\text{O}(\text{H}_2\text{O})(\text{LH})(\text{MeOH}))]$ [25]. The presence of a broadband at 3440 cm^{-1} due to $\nu(\text{OH})$ stretch and the appearance of a new medium intense band at 1135 cm^{-1} due to the $\delta(\text{Zr-OH})$ bending mode [26] also support the proposed structure of the present $\text{Zr}(\text{OH})_2(\text{IV})$ compound. $[\text{UO}_2(\text{LH})(\text{MeOH})]$ exhibits the $\nu_{\text{as}}(\text{O=U=O})$ stretch at 898 cm^{-1} and this band occurs in the usual range ($870\text{--}950\text{ cm}^{-1}$) observed for the majority of *trans*- $\text{UO}_2(\text{VI})$ compounds [27]. The force constant ($f_{\text{U-O}}$) and U-O bond length in the present dioxouranium(VI) compound are 6.70 mdyne/\AA and 1.74 \AA , respectively. These values are in the expected range ($6.58\text{--}7.03\text{ mdyne/\AA}$ and $1.60\text{--}1.92\text{ \AA}$) reported for the majority of $\text{UO}_2(\text{VI})$ compounds [26]. The new nonligand bands in the present coordination compounds in the low-frequency region are assigned to the $\nu(\text{M-O})$ ($550\text{--}570\text{ cm}^{-1}$) and the $\nu(\text{M-S})$ ($345\text{--}375\text{ cm}^{-1}$) and these bands [28] are in the expected order of increasing energy: $\nu(\text{M-S}) < \nu(\text{M-O})$.

3.2. NMR Spectral Studies. The NMR spectra of I and its coordination compounds were recorded in DMSO-d_6 . The chemical shifts (δ) are expressed in ppm downfield from TMS. The prominent resonance signals of these compounds were compared with the reported peaks [30]. I exhibits a singlet at $\delta 17.5\text{ ppm}$ due to the carboxylic proton, a sharp singlet at $\delta 13.60\text{ ppm}$ due to phenolic proton, a singlet at $\delta 2.35\text{ ppm}$ due to alcoholic proton, multiplets due to methylene protons at $\delta 4.70\text{--}4.79\text{ ppm}$, and multiplets between $\delta 7.34$ and 7.50 ppm due to the aromatic protons.

The occurrence of the resonance signal at the same frequency ($\delta 2.35\text{ ppm}$) due to alcoholic proton (CH_2OH) indicates the noninvolvement of the alcoholic group in coordination. The absence of the signal at $\delta 17.5\text{ ppm}$ due to the COOH proton in the coordination compounds indicates the deprotonation of the COOH group, followed by the involvement of its O atom in coordination. The absence of the resonance signal at $\delta 13.60\text{ ppm}$ due to the phenolic proton in the coordination compounds indicates the deprotonation of the phenolic OH group followed by its involvement in coordination [23]. The appearance of resonance signals at $2.81\text{--}2.85\text{ ppm}$ due to alcoholic proton and at $3.0\text{--}3.1\text{ ppm}$ due to methyl protons in the coordination compounds supports the presence of MeOH in these compounds.

3.3. Reactance Spectral Studies. The presence of three bands at 8700 , 13000 , and 18550 cm^{-1} due to ${}^4\text{T}_{1g}(\text{F})$, ${}^4\text{T}_{2g}(\text{v}_1)$, ${}^4\text{T}_{1g}(\text{F})$, ${}^4\text{A}_{2g}(\text{v}_2)$, and ${}^4\text{T}_{1g}(\text{F})$, ${}^4\text{T}_{1g}(\text{v}_3)$ transitions, respectively, suggests an octahedral arrangement of I around Co(II) ions in $[\text{Co}(\text{LH})(\text{MeOH})_3]$ [31]. The value of ν_3/ν_1 (2.13) lies in the range $2.00\text{--}2.80$, reported for most of the octahedral Co(II) compounds [30]. The spectral parameters [30–32] for $[\text{Co}(\text{LH})(\text{MeOH})_3]$ are $\text{Dq} = 979\text{ cm}^{-1}$, $\text{B}' = 729\text{ cm}^{-1}$, $\beta = \text{B}'/\text{B} = 0.75$, $\beta^0 = 25\%$, and $\text{CFSE} = -93.57\text{ kJ mol}^{-1}$. Reduction of Racah parameter from 971 cm^{-1} (free ion value) to 729 cm^{-1} and the β^0 value (25%) indicate the covalent nature of the compound and strong field nature of I [20]. $[\text{Ni}(\text{LH})(\text{MeOH})_3]$ shows three bands at 8980 , 15980 , and 24900 cm^{-1} due to ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{F})(\text{V}_1)$, ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})(\text{V}_2)$, and ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})(\text{V}_3)$ transitions, respectively, in an octahedral symmetry [31]. The value of V_2/V_1 is 1.78 which lies in the usual range ($1.60\text{--}1.82$), reported for the majority of octahedral Ni(II) compounds. The spectral parameters [31, 32] for $[\text{Ni}(\text{LH})(\text{MeOH})_3]$ are $\text{Dq} = 898\text{ cm}^{-1}$, $\text{B}' = 843\text{ cm}^{-1}$, $\beta = \text{B}'/\text{B} = 0.82$, $\beta^0 = 18\%$, and $\text{CFSE} = -128.75\text{ kJ mol}^{-1}$. The reduction of the Racah parameter from the free ion value (1030 cm^{-1}) to 843 cm^{-1} and the β^0 value (18%) are indicative of the presence of covalent nature of the compound and the strong field nature of the tridentate ligand (I) [19]. For a given ligand and given stereochemistry, the covalent character of the corresponding Co(II) and Ni(II) coordination compounds is comparable since Co(II) and Ni(II)



occupy the adjacent positions in the nephelauxetic metal ion series (i.e., Co(II) ~ Ni(II))^[19]. In the present Co(II) and Ni(II) coordination compounds, the β^0 values are quite comparable: [Co(LH)(MeOH)₃]: 25%; [Ni(LH)(MeOH)₃]: 18%. For a given ligand and given stereochemistry, the spectrochemical series of metal ions on the basis of increasing 10 Dq values is Ni(II) < Co(II). Our calculated 10 Dq values indicate that the 10 Dq values are in the expected order: Ni(II) < Co(II). The greater negative CFSE value (−128.75 kJ mol^{−1}) for [Ni(LH)(MeOH)₃] in comparison to that of [Co(LH)(MeOH)₃] (CFSE = −93.57 kJ mol^{−1}) is as expected [34].

3.4. Magnetic Measurements: The room temperature magnetic moments of the coordination compounds of I are presented in Table 2. The magnetic moments of [Co(LH)(MeOH)₃] and [Ni(LH)(MeOH)₃] are 4.75 and 3.14 B.M., respectively. These values are indicative of the magnetically dilute high-spin octahedral coordination compounds of Co(II) and Ni(II) ions^[35]. The coordination compounds of other ions are diamagnetic.

Conclusion

Thus, on the basis of analytical data, valence requirements, molecular weight, spectral and the magnetic studies, it is proposed that I behaves as a dibasic tridentate OOS donor ligand in the tetrahedral compound, [Cd(LH)(MeOH)] (II) and in the octahedral compounds, [M(LH)(MeOH)₃] [where M=Co(II), Ni(II)], (III), [Zr(OH)₂(LH)(MeOH)] (IV) and for [UO₂(LH)(MeOH)] (V), as shown in Figure 2.

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