

Physico-chemical and antibacterial studies on the coordination compounds of N-(2-carbamoylfuranyl)-C-(3'-carboxy-2'-hydroxyphenyl) thiazolidin-4-one

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An equimolar mixture of 3-formylsalicylic acid and furoic acid hydrazide, refluxed in MeOH, forms the Schiff base (**I**). The latter upon reacting with mercaptoacetic acid in dry benzene, undergoes cyclization and forms N-(2-carbamoylfuranyl)-C-(3'-carboxy-2'-hydroxyphenyl)thiazolidin-4-one, LH₃ (**II**). A MeOH solution of **II** reacts with Co(II), Ni(II), Zn(II), Fe(III) and MoO₂(VI) ions and forms the coordination compounds [M(LH)(MeOH)₂] [where M = Co(II), Ni(II)], [Zn(LH)], [FeCl₃(LH₃)] and [MoO₂(acac)(LH₂)] (here acacH = pentane-2,4-dione). The coordination compounds were characterized on the basis of elemental analyses, molar conductance, molecular weight, spectral (IR, reflectance, NMR) studies and magnetic susceptibility measurements. All coordination compounds are non-electrolytes ($\Lambda_M = 2.8-11.7 \text{ mho cm}^2 \text{ mol}^{-1}$) in DMF. **II** behaves as a monobasic bidentate OS donor ligand in [MoO₂(acac)(LH₂)], neutral tridentate ONO donor ligand in [FeCl₃(LH₃)], and dibasic tetradentate OONO donor ligand in [M(LH)(MeOH)₂] [where M = Co(II), Ni(II)] and [Zn(LH)]. All compounds are monomers in diphenyl. The absolute coordination number of Zn(II) ion is 4 and those of Co(II), Ni(II), Fe(III) and MoO₂(VI) are 6. Tetrahedral structure for [Zn(LH)] and octahedral structure for the rest of the compounds are proposed. The ligand (**II**) and its compounds show antibacterial activities towards *E. Coli* (Gram Negative) and *S. Aureus* (Gram positive).

Keywords: thiazolidin-4-one, coordination compounds, monomeric, magnetic susceptibility, complexometric titration, strong field, covalent character.

INTRODUCTION

Thiazolidinones are derivatives of thiazolidine belonging to an important group of heterocyclic compounds. 4-Thiazolidinone, with a carbonyl group at 4th position is a topic of numerous reports concerning its chemistry and applications. The presence of a thiazolidine ring in penicillin and related derivatives was the first recognition of its occurrence in nature [1]. These heterocyclic compounds containing S and N as heteroatoms may bear imine or carboxyl groups. Thiazolidine possesses two donor sites situated at the =NH group and the sulphur atom of the ring, whereas for its derivatives additional coordination sites appear. All above mentioned donor atoms are responsible for the formation of various complexes with transition metal ions [2]. Due to their pharmaceutical importance, thiazolidin-4-ones have played an important role in medicinal chemistry. They have been extensively studied because of their biological activities such as antioxidant [3], antitubercular [4], antimicrobial [5], antibacterial [6], anticonvulsant

[7], antifungal [8] etc.

Metal complexes play an important role in plant and animal life due to their physico-chemical and biological properties. Organosulphur compounds, in the form of their metal complexes, exhibit a wide range of biological properties. Most of the drugs have improved pharmacological properties in the form of metal complexes. Transition metal based drugs are more effective than the uncomplexed drugs [9]. These facts motivated us to study the coordination behaviour of newly synthesized thiazolidinone complexes with some transition metals.

A perusal of the literature indicates that relatively little work has been carried out on the coordination compounds of thiazolidinones [10,11] and there is no report on the coordination compounds of N-(2-carbamoylfuranyl)-C-(3'-carboxy-2'-hydroxyphenyl)thiazolidin-4-one, LH₃ (**II**) as shown in Figure-1 and its coordination compounds with Co(II), Ni(II), Zn(II), Fe(III) and MoO₂(VI) ions. These compounds are very well coordinated and biologically active.

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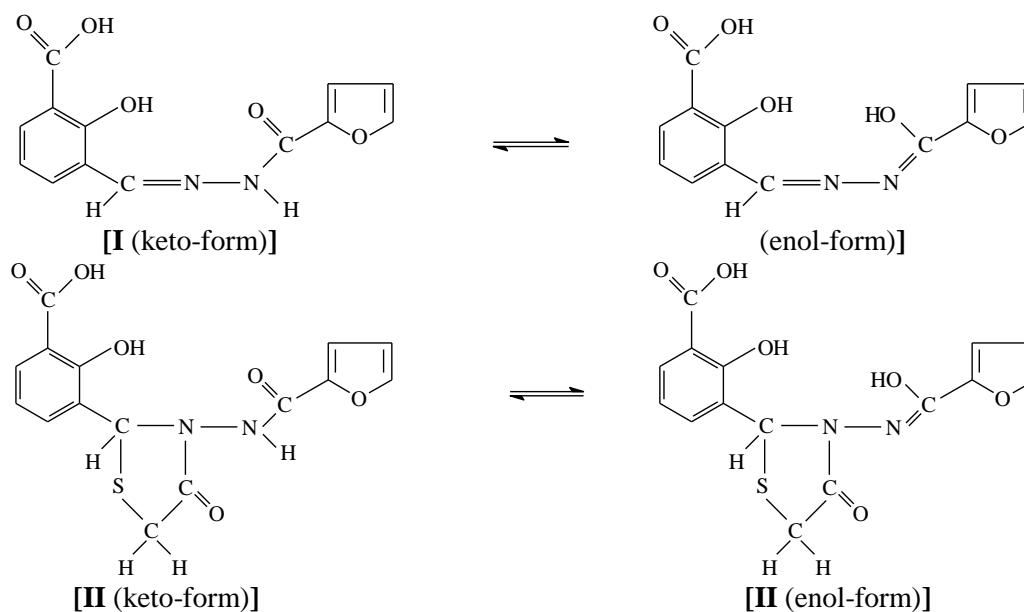


Fig.1

EXPERIMENTAL

Materials

Furoic acid hydrazide [Aldrich]; nickel (II) acetate tetrahydrate, iron (III) chloride (anhydrous) [Sarabhai]; zinc(II) acetate dihydrate [SD's Fine]; cobalt (II) acetate tetrahydrate, ammonium molybdate(VI) tetrahydrate, methanol, ethanol, mercaptoacetic acid, dry benzene, sodium bicarbonate [Ranbaxy] were used as supplied for the syntheses. 3-Formylsalicylic acid and bis(acetylacetonato)dioxomolybdenum(VI) were synthesized following the reported procedures [12].

Analyses and Physical Measurements

The organic skeleton of the respective coordination compounds was decomposed by slow heating of ~ 0.1 g of the latter with conc. HNO_3 . The residue was dissolved in a minimum amount of conc. HCl and the corresponding metal ions were determined as follows: the Ni(II) content of the coordination compound was determined by complexometric titration with standardized EDTA solution using murexide as the indicator. The Co(II) and Zn(II) contents in the respective coordination compounds were determined by complexometric titration with standardized EDTA solution using xylenol orange as an indicator. The Fe(III) ions were reduced to Fe(II) ions with aqueous SnCl_2 and then determined with a standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution using N-phenylanthranilic acid as an indicator. The molybdenum content was estimated gravimetrically after decomposing the given $\text{MoO}_2(\text{VI})$ compound with a few drops of conc. HNO_3 and conc. H_2SO_4 and then igniting the residue in an electric Bunsen burner at 500°C . MoO_3 obtained was dissolved in

6N NaOH and then molybdenum was determined as bis (8-hydroxyquinolinato)dioxomolybdenum (VI). The C, H and N contents of LH_3 and its coordination compounds were determined on a CHN Eager analyzer model-300. The S and Cl contents were determined gravimetrically as BaSO_4 and AgCl , respectively. The molecular weight measurements were carried out by the Rast method using diphenyl as the solvent [13]. The molar conductances (Λ_M) of the coordination compounds were measured in DMF with the help of a Toshniwal conductivity bridge (CL01-02A) and a dip type cell calibrated with KCl solutions. The IR spectra were recorded in KBr pellets ($4000\text{--}400\text{ cm}^{-1}$) on a Beckman-20 spectrophotometer. The reflectance spectra were recorded on a Beckmann DU spectrophotometer with a reflectance arrangement. The magnetic susceptibility measurements were carried out at room temperature using $\text{Hg}[\text{Co}(\text{NCS})_4]$ as the standard [14]. The diamagnetic corrections were computed using Pascal's constants. The magnetic susceptibilities were corrected for the temperature independent paramagnetism term (TIP) [14] using a value of 200×10^{-6} cgs units for Ni(II) and Co(II), and zero for Fe(III) ions.

Synthesis of the Schiff base (I)

The title compound was synthesized following the published procedure [15].

Synthesis of N-(2-carbamoylfuranyl)-C-(3'-carboxy-2'-hydroxyphenyl)thiazolidin-4-one (II)

A dry benzene solution of I (2.74 g, 10 mmol) and mercaptoacetic acid (0.92 g, 10 mmol) was refluxed for 12 h on a water bath. The mixture was

cooled to room temperature and then washed with 10% sodium bicarbonate solution. The benzene layer was separated using a separating funnel. The evaporation of the excess of solvent gave a solid product which was filtered, washed with benzene and recrystallized from petroleum ether. The compound was dried *in vacuo* at room temperature over silica gel. Yield = 17%. *Anal*: (**II**, C₁₅H₁₂N₂O₆S) (obsd: C, 51.42%; H, 3.34%; N, 8.17%; S, 9.35%. calcd.: C, 51.72%; H, 3.45%; N, 8.04%; S, 9.20%); IR bands (KBr): 2850 cm⁻¹ [$\nu(\text{O}-\text{H})$] (intramolecular H-bonding), 1695 cm⁻¹ [$\nu(\text{C}=\text{O})$] (thiazolidinone ring), 1670 cm⁻¹ [$\nu(\text{C}=\text{O})$] (carboxylic), 1645 cm⁻¹ [$\nu(\text{C}=\text{O})$] (amide), 1565 cm⁻¹ [$\nu(\text{C}-\text{N})$] (thiazolidinone ring), 1532 cm⁻¹ [$\nu(\text{C}-\text{O})$] (phenolic), 1090 cm⁻¹ [$\nu(\text{C}-\text{O}-\text{C})$] (furan ring) and 825 cm⁻¹ [$\nu(\text{C}-\text{S})$] (thiazolidinone ring).

Syntheses of the coordination compounds of **II**

A MeOH solution (30-50 mL) of the appropriate metal salt (10 mmol) was added to a MeOH solution (50 mL) of **II** (3.48 g, 10 mmol) and the mixture was then refluxed for 3-4 h. The solid products formed were suction filtered, washed with MeOH and were then dried as mentioned above. Yield = 30–60%.

RESULTS AND DISCUSSION

The Schiff base **I** in a dry benzene solution reacts with mercaptoacetic acid and forms N-(2-carbamoylfuranyl)-C-(3'-carboxy-2'-

hydroxyphenyl) thiazolidin-4-one, LH₃ (**II**). The reaction of the latter with appropriate metal salts in 1:1 molar ratio in MeOH produces the coordination compounds [M(LH)(MeOH)₂] [where M = Co(II), Ni(II)], [Zn(LH)], [FeCl₃(LH₃)] and [MoO₂(acac)(LH₂)] (here acacH = pentane-2,4-dione). The compounds are stable in air at room temperature. They are insoluble in H₂O, partially soluble in MeOH, EtOH and completely soluble in DMSO and DMF. The molar conductance measurements ($\Lambda_M = 2.8-11.7$ mho cm² mol⁻¹) indicate their non-electrolytic nature. The analytical data of **II** and its coordination compounds are given in Table 1.

Infrared Spectral Studies

The infrared spectra of **II** and its coordination compounds were recorded in KBr and the prominent peaks (in cm⁻¹) are shown in Table 2. **I** exhibits a $\nu(\text{C}=\text{N})$ (azomethine) stretch at 1630 cm⁻¹. This band disappears in **II** and a new band appears at 1565 cm⁻¹ due to the $\nu(\text{C}-\text{N})$ (thiazolidinone ring) stretch [16] indicating the conversion of **I** into **II**. The formation of **II** is further supported by the appearance of a new band at 825 cm⁻¹ due to the $\nu(\text{C}-\text{S})$ (thiazolidinone ring) stretch [17]. The $\nu(\text{C}-\text{N})$ (thiazolidinone ring) stretch of **II** shifts to lower energy by 50-72 cm⁻¹ in the coordination compounds of Co(II), Ni(II) and Zn(II) ions suggesting the involvement of the ring N atom towards coordination [17].

Table 1. Analytical, molar conductance (Λ_M) and molecular weight data of **II** and its coordination compounds.

Compound	Mol. formula	Λ_M (mho cm ² mol ⁻¹)	Mol. wt obsd (calcd)	obsd(calcd)%					
				M	C	H	N	S	Cl
LH ₃ (II)	C ₁₅ H ₁₂ N ₂ O ₆ S	–	348.0 ^a (348.0)	–	51.42 (51.72)	3.34 (3.45)	8.17 (8.04)	9.35 (9.20)	–
[Co(LH)(MeOH) ₂]	CoC ₁₇ H ₁₈ N ₂ O ₈ S	5.3	464.9 ^b (468.9)	12.51 (12.56)	43.23 (43.51)	3.77 (3.84)	5.89 (5.97)	6.60 (6.82)	–
[Ni(LH)(MeOH) ₂]	NiC ₁₇ H ₁₈ N ₂ O ₈ S	2.8	465.4 ^b (468.7)	12.40 (12.52)	43.22 (43.52)	3.74 (3.84)	5.88 (5.97)	6.91 (6.83)	–
[Zn(LH)]	ZnC ₁₅ H ₁₀ N ₂ O ₆ S	8.2	427.2 ^b (411.4)	15.69 (15.92)	43.82 (43.74)	2.51 (2.43)	6.68 (6.81)	7.96 (7.78)	–
[FeCl ₃ (LH ₃)]	FeC ₁₅ H ₁₂ N ₂ O ₆ SCl ₃	11.7	528.1 ^b (510.5)	10.72 (10.97)	34.98 (35.26)	2.19 (2.35)	5.66 (5.48)	6.05 (6.27)	20.50 (20.86)
[MoO ₂ (acac)(LH ₂)]	MoC ₂₀ H ₁₈ N ₂ O ₁₀ S	7.3	558.1 ^b (573.9)	16.54 (16.71)	41.61 (41.82)	3.16 (3.14)	4.73 (4.88)	5.51 (5.58)	–

Abbreviation; ^aMass spectral data, ^bRast method data

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

Compound	$\nu(\text{C-N})$ (thiazolidinone)	ν (C-O) ϕ	ν_{as} (COO)	ν_{s} (COO)	$\nu(\text{C=O})$ (amide)	$\nu(\text{C-O})$ (MeOH)	ν_{max}	Mag. moment (B. M.)
LH ₃ (II)	1565	1532	–	–	1645	–	–	Diamagnetic
[Co(LH)(MeOH) ₂]	1515	1542	1566	1348	1628	976	8900,13290, 19400	4.78
[Ni(LH)(MeOH) ₂]	1500	1541	1568	1358	1620	970	9460,17215, 26000	3.26
[Zn(LH)]	1493	1542	1560	1350	1611	–	–	Diamagnetic
[FeCl ₃ (LH ₃)]	1565	1532	–	–	1645	–	13150, 16900, 24750	5.97
[MoO ₂ (acac)(LH ₂)]	1565	1541	–	–	1645	–	–	Diamagnetic

However, the existence of this band at the same energy in the coordination compounds of Fe(III) and MoO₂(VI) ions indicates the non-involvement of the ring N atom towards coordination. **II** exhibits a strong band at 2850 cm⁻¹ due to the intramolecular H-bonded OH groups of phenolic and/or carboxylic acid moieties [18]. The appearance of this band at almost the same energy in [FeCl₃(LH₃)] is indicative of the non-involvement of the OH groups of the above moieties towards coordination. On the other hand, the coordination compounds of the other metal ions do not show this band indicating the breakdown of H-bonding and subsequent deprotonation of the OH groups followed by the involvement of phenolic and carboxylic O atoms in coordination. The $\nu(\text{C=O})$ (thiazolidinone ring) stretch [19] of **II** occurs at 1695 cm⁻¹. This band shifts to 1660 cm⁻¹ in [FeCl₃(LH₃)] indicating coordination through the O atom of the carbonyl group of the thiazolidinone moiety. However, the above band remains unchanged in the rest of the coordination compounds suggesting the non-involvement of the above O atom in coordination. The $\nu(\text{C=O})$ (carboxylic) stretch [20] of **II** occurs at 1670 cm⁻¹. This band remains unchanged in the coordination compounds of Fe(III) and MoO₂(VI) ions indicating the non-involvement of the O atom of the carboxylic group in coordination. However, the remaining coordination compounds display two new bands between 1560-1568 cm⁻¹ and 1348-1358 cm⁻¹. These bands are assigned to the $\nu_{\text{as}}(\text{COO})$ and $\nu_{\text{s}}(\text{COO})$ stretches of the carboxylato ligand, respectively. The energy separation ($\Delta\nu = 210\text{-}218$ cm⁻¹) between the $\nu_{\text{as}}(\text{COO})$ and $\nu_{\text{s}}(\text{COO})$ stretches is indicative of the monodentate nature of the carboxylato ligand [21]. The presence of a strong band at 1645 cm⁻¹ due to the $\nu(\text{C=O})$ (amide) stretch in **II** indicates that it occurs in the keto-form [12]. This band shifts to lower energy by 17-34 cm⁻¹ in the coordination compounds of Co(II), Ni(II) and Zn(II) ions, suggesting the involvement of the keto O atom in coordination. However, the

existence of this band at the same energy in the coordination compounds of Fe(III) and MoO₂(VI) ions indicates the non-involvement of the keto O atom in coordination. The $\nu(\text{C-O})\phi$ stretch [22] (1532 cm⁻¹) of **II** undergoes a positive shift by ≤ 10 cm⁻¹ in the coordination compounds of Co(II), Ni(II), Zn(II) and MoO₂(VI) ions. This band remains unchanged in the Fe(III) compound indicating the non-involvement of phenolic O in coordination. The $\nu(\text{C-S})$ (thiazolidinone ring) stretch [16] of **II** shifts from 825 cm⁻¹ to lower energy by 42 cm⁻¹ in [MoO₂(acac)(LH₂)] suggesting the formation of a bond between the metal ions and the S atom. However, it remains unchanged in the rest of the coordination compounds indicating non-involvement of S atom in coordination. The [$\nu(\text{C-O-C})$ (furan moiety)] stretch of **II** occurs at 1090 cm⁻¹. The appearance of a band at the same energy in the coordination compounds of Co(II), Ni(II), Zn(II), and MoO₂(VI) ions indicates the non-involvement of the O atom of the furan moiety in coordination. On the other hand, the negative shift by 60 cm⁻¹ of this band in [FeCl₃(LH₃)] indicates the coordination through the O atom of the furan moiety. The presence of a broad band between 3300-3400 cm⁻¹ due to $\nu(\text{O-H})$ (MeOH) stretch and the decrease in $\nu(\text{C-O})$ (MeOH) stretch [23] from 1034 cm⁻¹ to lower energy by 58-64 cm⁻¹ in the coordination compounds of Co(II) and Ni(II) ions indicate the involvement of the O atom of MeOH in coordination [23]. The absence of a band between 820-860 cm⁻¹ in [FeCl₃(LH₃)] precludes the presence of a $\nu(\text{Fe-O-Fe})$ bridged structure [24]. The molecular weight data (Table 1) also suggest the same. The appearance of two new bands due to the $\nu_{\text{s}}(\text{O=Mo=O})$ stretch (940 cm⁻¹) and the $\nu_{\text{as}}(\text{O=Mo=O})$ stretch (910 cm⁻¹) in [MoO₂(acac)(LH₂)] are indicative of a *cis*-MoO₂ configuration [20]. The new non-ligand bands in the present coordination compounds in the low frequency region are assigned to $\nu(\text{M-O})$ (538-563 cm⁻¹), $\nu(\text{M-N})$ (418-456 cm⁻¹) and $\nu(\text{M-S})$ (342-369 cm⁻¹) vibrations [25].

NMR Spectral Studies

The NMR spectra of **II** and [Zn(LH)] 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 [26]. **II** exhibits a singlet at δ 17.5 ppm due to the carboxylic proton, a sharp singlet at δ 13.61 ppm due to phenolic proton, a singlet at δ 10.8 ppm (due to CONH proton) and at δ 11.3 ppm (due to HO-C=N, tautomeric protons), and multiplets between δ 7.36-7.71 ppm due to aromatic protons. The absence of the signal at δ 17.5 ppm due to the COOH proton in [Zn(LH)] indicates the deprotonation of the COOH group, followed by the involvement of its O atom in coordination. The absence of the resonance signal at δ 13.61 ppm due to the phenolic proton in [Zn(LH)] indicates the deprotonation of the phenolic OH group followed by its involvement in coordination [27]. The singlet at δ 10.4 ppm due to CONH proton remains unchanged in the complex which shows involvement of keto oxygen in the coordination.

Reflectance Spectral Studies

The presence of three bands at 8900, 13290 and 19400 cm^{-1} due to ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(v_1)$, ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(v_2)$ and ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(v_3)$ transitions, respectively, in [Co(LH)(MeOH)₂] suggests an octahedral arrangement of **II** around the Co(II) ions [28]. The v_3/v_1 value is 2.18 which lies in the usual range (2.00-2.80) reported for the majority of octahedral Co(II) coordination compounds [28]. The parameters are: $Dq = 1004 \text{ cm}^{-1}$, $B' = 776 \text{ cm}^{-1}$, $\beta = B'/B = 0.80$, $\beta^0 = 20\%$ and $\text{CFSE} = -96.13 \text{ kJ mol}^{-1}$. The reduction of Racah parameter from the free ion value of 971 cm^{-1} to 776 cm^{-1} and the β^0 value (20%) indicate the covalent nature of the compound and the strong field nature of the ligand [14]. [Ni(LH)(MeOH)₂] exhibits three bands at 9460, 17215 and 26000 cm^{-1} due to ${}^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)(v_1)$, ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)(v_2)$ and ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)(v_3)$ transitions, respectively, in an octahedral symmetry [29]. The v_2/v_1 value is 1.81 and it lies in the usual range (1.60-1.82) reported for the majority of octahedral Ni(II) coordination compounds [28]. Here the values of the spectral parameters are: $Dq = 946 \text{ cm}^{-1}$, $B' = 870 \text{ cm}^{-1}$, $\beta = B'/B = 0.84$, $\beta^0 = 16\%$ and $\text{CFSE} = -124.73 \text{ kJ mole}^{-1}$. The reduction of the Racah parameter from the free ion value (1030 cm^{-1}) to 870 cm^{-1} and the β^0 value (16%) are indicative of the covalent nature of the compound and the strong field nature of the ligand [14]. 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)]. In the present Co(II) and Ni(II) coordination compounds, the β^0 values are quite comparable: [Co(LH)(MeOH)₂]: 20%; [Ni(LH)(MeOH)₂]: 16%. For a given ligand and given stereochemistry, the spectrochemical series of metal ions on the basis of increasing $10Dq$ values is [14]: Ni(II) < Co(II). Our calculated $10Dq$ values indicate that the $10Dq$ values are in the expected order: Ni(II) < Co(II). The greater negative CFSE value ($-124.73 \text{ kJ mol}^{-1}$) for [Ni(LH)(MeOH)₂] in comparison to that [Co(LH)(MeOH)₂] ($\text{CFSE} = -96.13 \text{ kJ mol}^{-1}$) is as expected [29]. The coordination compound [FeCl₃(LH₃)] exhibits three bands at 13150, 16900 and 24750 cm^{-1} due to ${}^6A_{1g} \rightarrow {}^4T_{1g}(G)$, ${}^6A_{1g} \rightarrow {}^4T_{2g}(G)$ and ${}^6A_{1g} \rightarrow {}^4A_{1g}(G)$ transitions, respectively, in an octahedral environment [30].

Magnetic Measurements

The magnetic moments of [Co(LH)(MeOH)₂], [Ni(LH)(MeOH)₂] and [FeCl₃(LH₃)] are 4.78, 3.26 and 5.97 B.M., respectively. These values are in the normal ranges reported for magnetically diluted octahedral compounds of Co(II), Ni(II) and Fe(III) ions [31]. The coordination compounds of Zn(II) and MoO₂(VI) ions of **II** are diamagnetic, as expected.

Antibacterial Studies

The antibacterial activity of ligand (**II**) and its complexes was tested against bacteria, *E. Coli* (Gram negative) and *S. aureus* (Gram positive) by the disc diffusion method (Table 3).

Table 3. Antibacterial activity of **II** and its coordination compounds (zone of inhibition in mm)

Compound	<i>E. Coli</i> (Gram negative)	<i>S. Aureus</i> (Gram positive)
LH ₃ (II)	7	6
[Co(LH)(MeOH) ₂]	8	7
[Ni(LH)(MeOH) ₂]	7	10
[Zn(LH)]	10	12
[FeCl ₃ (LH ₃)]	8	9
[MoO ₂ (acac)(LH ₂)]	11	10

Stock solutions were prepared by dissolving the compounds in DMSO. Under aseptic conditions, plain sterilised discs were soaked in the solutions of the compounds overnight. Test culture was spread over the plates containing Mueller Hinton Agar

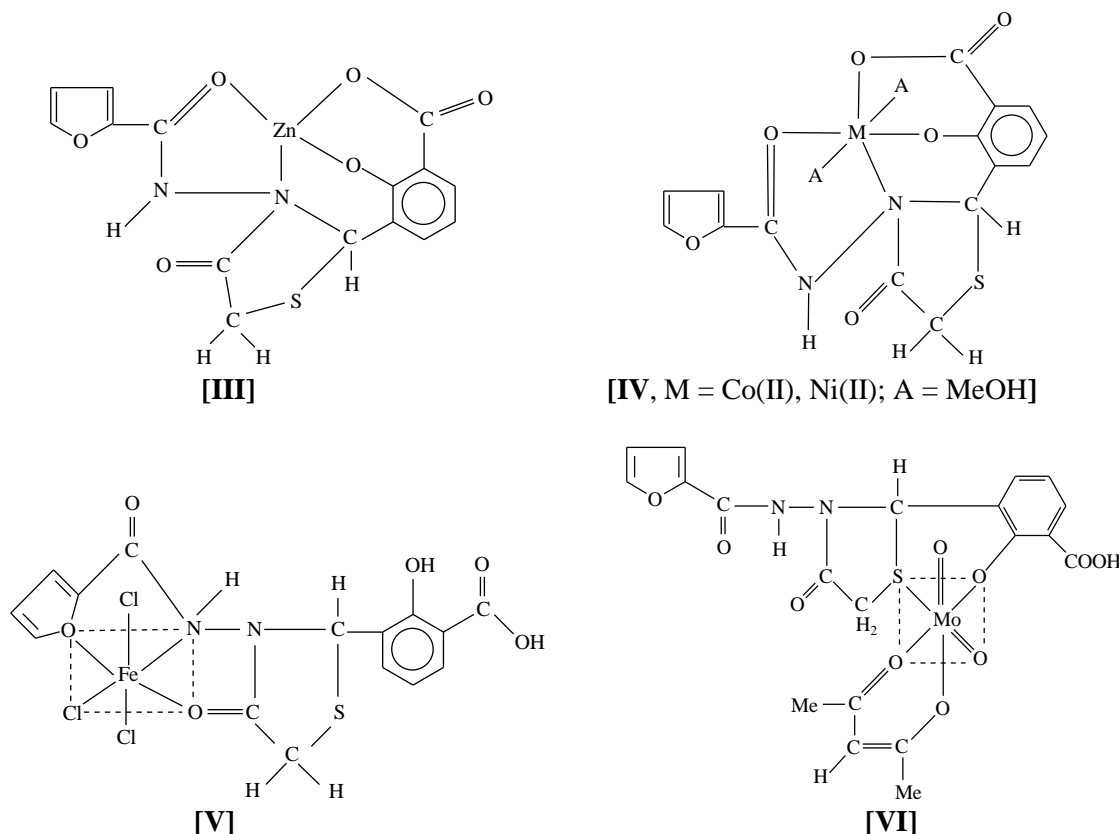


Fig. 2.

(MHA) using a sterile swab. Inoculated plates were dried for 30 minutes and discs were placed on the inoculated plates. The plates were left for 30 min at room temperature to allow diffusion. The plates were then incubated at 37 °C for 24 h for *E. Coli* and *S. aureus*. After incubation, the diameter of the zone of inhibition was noted for each disc.

Determination of Minimum Inhibitory Concentration (MIC)

The stock solutions of the compounds were prepared using distilled water as diluent. In a set of test tubes containing 2 mL of Mueller Hinton Broth, the compounds were serially diluted. 2 mL of the test culture was added to all tubes and the latter were incubated at 37°C for 24 h. Lack of turbidity was noted for the determination of MIC (Table 4).

Table 4. Minimum inhibitory concentration (MIC) of II and its coordination compounds ($\mu\text{g/ml}$)

Compound	<i>E. Coli</i> (Gram negative)	<i>S. Aureus</i> (Gram positive)
LH ₃ (II)	64	32
[Co(LH)(MeOH) ₂]	32	64
[Ni(LH)(MeOH) ₂]	64	64
[Zn(LH)]	64	32
[FeCl ₃ (LH ₃)]	32	32
[MoO ₂ (acac)(LH ₂)]	32	64

CONCLUSIONS

From the ongoing discussion it may be stated that **II** behaves as a monobasic bidentate OS donor ligand in [MoO₂(acac)(LH₂)], neutral tridentate ONO donor ligand in [FeCl₃(LH₃)] and dibasic tetradentate OONO donor ligand in [M(LH)(MeOH)₂] [where M = Co(II), Ni(II)] and [Zn(LH)]. Thus, on the basis of analytical, molecular weight, spectral and magnetic studies, we propose a tetrahedral structure, **III** for [Zn(LH)] and an octahedral structure, **IV** for [M(LH)(MeOH)₂] [where M = Co(II), Ni(II); A = MeOH], **V** for [FeCl₃(LH₃)] and **VI** for [MoO₂(acac)(LH₂)] as shown in Figure-2.

The newly synthesized compounds represent a class of coordination compounds of thiazolidinone and prove themselves as potential compounds in terms of coordination chemistry and biological activities.

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ФИЗИКОХИМИЧНИ И АНТИБАКТЕРИАЛНИ ИЗСЛЕДВАНИЯ ВЪРХУ КООРДИНАЦИОННИ СЪЕДИНЕНИЯ С N-(2-КАРБАМОИЛ)-С-(3'-КАРБОКСИ-2'- ХИДРОКСИФЕНИЛ) ТИАЗОЛИДИН-4-ОН

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(Резюме)

Еквимоларната смес от 3-формил-салицилова киселина и хидразида на фуриевата киселина, разтворени в метанол образува Шифова база (I). Последната претърпява циклизация и образува N-(2-карбамоил-фураил)-С-(3'-карбокси-2'-хидроксифенил) тиазолидин-4-он, LH₃ (II) при реакцията с меркаптооцетна киселина в сух бензен. Разтворът на II в метанол реагира с йоните Co(II), Ni(II), Zn(II), Fe(III) и MoO₂(VI) и се образуват координационни съединения от типа [M(LH)(MeOH)₂] [където M = Co(II), Ni(II)], [Zn(LH)], [FeCl₃(LH₃)] и [MoO₂(асас)(LH₂)] (тук асасН = pentane-2,4-dione). Координационните съединения са охарактеризирани на базата на елементен анализ, моларна проводимост, молекулно тегло, спектрални методи (IR, отражение, NMR) и измервания на магнитния susceptibilitet. Всички координационни съединения не са електролити ($\Lambda_m = 2.8-11.7 \text{ Ohm cm}^2 \text{ mol}^{-1}$) в DMF. II се отнася като монобазичен бидендатен OS донорен лиганд в [MoO₂(асас)(LH₂)], неутрален тридендатен ONO донорен лиганд в [FeCl₃(LH₃)], и дибазичен тетрадендатен OONO донорен лиганд в [M(LH)(MeOH)₂] [където M = Co(II), Ni(II)] и [Zn(LH)]. Всички съединения са мономери в дифенил. Абсолютното координационно число на Zn(II)-йоните е 4, а тези на Co(II), Ni(II), Fe(III) и MoO₂(VI) са 6. Предлага се тетраедрична структура на [Zn(LH)] и октаедрична структура за останалите съединения. Лигандът (II) и неговите съединения показват антибактериални свойства спрямо *E. Coli* (Грам-отрицателни) и *S. aureus* (Грам-положителни).