

Research Article

Physico-Chemical Studies on the Coordination Compounds of Thiazolidin-4-One

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A dry benzene solution of the Schiff base, *N*-(2-hydroxyphenyl)-3'-carboxy-2'-hydroxybenzylideneimine upon reacting with mercaptoacetic acid undergoes cyclization and forms *N*-(2-hydroxyphenyl)-C-(3'-carboxy-2'-hydroxyphenyl)thiazolidin-4-one, LH₃ (I). A MeOH solution of I reacts with Mn^{II}, Cu^{II}, Zn^{II}, Fe^{III} and MoO₂^{VI} ions and forms the monomeric coordination compounds, [Mn(LH)(MeOH)₃] (II), [Cu(LH)(MeOH)] (III), [Zn(LH)(MeOH)] (IV), [FeCl(LH)(MeOH)₂] (V), and [MoO₂(LH)(MeOH)](VI). The coordination compounds have been characterized on the basis of elemental analyses, molar conductance, molecular weight, spectral (IR, reflectance, ESR) studies, and magnetic susceptibility measurements. I behaves as a dibasic tridentate OOS donor ligand in these compounds. The compounds are nonelectrolytes ($\Lambda_M = 6.2\text{--}13.8 \text{ mho cm}^2 \text{ mol}^{-1}$) in DMF. A square-planar structure for III; a tetrahedral structure for IV and an octahedral structure for II, V, and VI are suggested.

1. Introduction

Heterocyclic compounds of Schiff bases possessing thiazolidin-4-one skeleton (having carbonyl group at 4th position) are known for their versatile pharmacological and industrial importance [1]. They have been studied extensively because of their ready accessibility, diversified chemical reactivity, and broad spectrum of biological activities [2].

In recent years, thiazolidin-4-ones have been investigated most extensively. The presence of a thiazolidine ring in penicillin and related derivatives was the first recognition of its occurrence in nature [3]. Thiazolidine-4-one represents a prevalent scaffold in drug discovery [4]. Literature surveys show that thiazolidin-4-ones have many interesting activity profiles, namely COX-1 inhibitors [5], inhibitors of the bacterial enzyme MurB, which was a precursor acting during the biosynthesis of peptidoglycan [6], nonnucleoside inhibitors

of HIV-RT [7] and antihistaminic agents [8]. Thiazolidinones are also known to exhibit antibacterial, anticonvulsant, antifungal, amoebicidal, antioxidant, and anticancer [9–12] activities.

A perusal of the literature reveals that much has been reported on the syntheses and characterization [13, 14] of a variety of thiazolidin-4-ones, but little is known about their coordination compounds [15–17].

Metal ions play a key role in the actions of drugs. They are involved in specific interactions with antibiotics, proteins, membrane components, nucleic acids, and other biomolecules [18, 19]. Many drugs possess modified pharmacological properties in the form of the metal complexes. Transition metal ions possess an important role in the design of metal-based drugs and such complexes are more effective against infectious diseases compared to the uncomplexed drugs [20, 21]. Metals complexes of Cu(II) has proved to be beneficial in diseases such as tuberculosis, gastric ulcers,

rheumatoid arthritis, and cancers [22]. The complexes of Fe(III) and Mn(II) also have excellent medicinal values [23].

Hence these facts prompted us to explore the coordination behavior of a newly synthesized thiazolidin-4-one with transition metal ions.

In this paper, we describe the syntheses and characterization of *N*-(2-hydroxyphenyl)-*C*-(3'-carboxy-2'-hydroxyphenyl) thiazolidin-4-one, LH₃ (**I**) and its coordination compounds with Mn^{II}, Cu^{II}, Zn^{II}, Fe^{III}, and MoO₂^{VI} ions. The structure of Schiff base and thiazolidin-4-one, LH₃ (**I**) is shown in Figure 1.

2. Experimental

2.1. Materials. 2-Aminophenol [Loba-Chemie (Mumbai)], mercaptoacetic acid, dry benzene, sodium bicarbonate, ammonium molybdate(VI) tetrahydrate, acetyl acetone [Ranbaxy], manganese(II) acetate tetrahydrate, iron(III) chloride (anhydrous) [Sarabhai], copper(II) acetate monohydrate, and zinc(II) acetate dihydrate [s d fine-chem Limited] were used as received for the syntheses. 3-Aldehyde-2-hydroxybenzoic acid and bis(acetylacetonato) dioxomolybdenum(VI) were synthesized by following the reported procedure [24].

2.2. Analyses and Physical Measurements. The organic skeleton of the respective coordination compounds was decomposed by the slow heating of ~0.1 g of the latter, with conc. HNO₃. The residue was dissolved in minimum amount of conc. HCl and the corresponding metal ions were estimated as follows: the Mn(II) and Zn(II) contents of the respective coordination compounds were estimated by complexometric titration method against standardized EDTA solution using eriochrome black-T as the indicator. The Cu(II) contents was estimated iodometrically against a standard solution of sodium thiosulphate to the starch end point. The Fe(III) ions were reduced to Fe(II) ions with aqueous SnCl₂ and then estimated against standard K₂Cr₂O₇ solution using *N*-phenylanthranilic acid as an indicator. The molybdenum contents was estimated gravimetrically after decomposing the given MoO₂(VI) compound with a few drops of conc. HNO₃ and conc. H₂SO₄, and then igniting the residue in an electric Bunsen at 500°C. MoO₃ obtained was dissolved in 6N NaOH, and then molybdenum was estimated as bis(8-hydroxyquinolinato) dioxomolybdenum(VI).

The C, H, and N contents of **I** and its coordination compounds were determined by CHN Eager analyzer model-300. The S and Cl contents of **I** and its coordination compounds were estimated gravimetrically as BaSO₄ and AgCl, respectively. The molecular weight measurements were carried out by the Rast method using diphenyl as the solvent [25].

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 (4000–400 cm⁻¹) on a Beckman-20 spectrophotometer. The reflectance spectra were recorded on a Beckmann DU

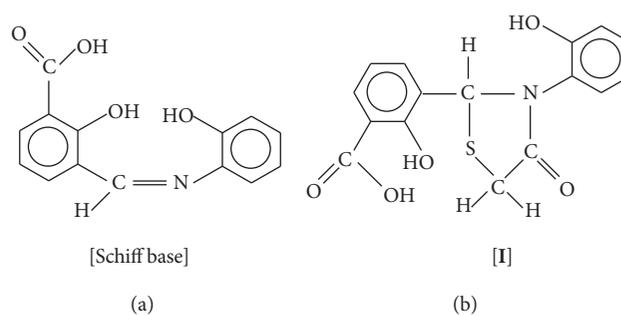


FIGURE 1

spectrophotometer attached with a reflectance arrangement. The magnetic susceptibility measurements were carried out at room temperature, using Hg[Co(NCS)₄] as the standard [26]. The diamagnetic corrections were computed using Pascal's constants. The magnetic susceptibilities were corrected for temperature independent paramagnetism term (TIP) [26] using value of 60×10^{-6} cgs units for Cu(II) ions, zero for Mn(II), and Fe(III) ions.

The ESR spectrum of **III** was recorded at liquid N₂ temperature in polycrystalline solids on a Varian V4502-12 X-band ESR spectrophotometer with 100 KHz modulation using diphenylpicrylhydrazide as a *g*-marker and monitoring the frequency with frequency meter.

2.3. Synthesis of the Schiff Base. The Schiff base was prepared as per the reported procedure [27].

2.4. Synthesis of I. A dry benzene solution of the Schiff base (2.57 g, 10 mmol) and mercaptoacetic acid (0.92 g, 10 mmol) were refluxed for 12 h on a water bath. The mixture was cooled to room temperature and then was washed with 10% sodium bicarbonate solution. The benzene layer was separated using a separating funnel. The partial evaporation of the benzene layer gave a solid product, which was filtered, washed with, and recrystallized from petroleum ether. Yield = 25%. *Anal.* (**I**, C₁₆H₁₃NO₅S) (obsd: C, 57.80%; H, 3.75%; N, 4.10%; S, 9.50%. calc.: C, 58.01%; H, 3.93%; N, 4.23%; S, 9.67%); IR bands (KBr): 2860 cm⁻¹ [ν (O-H) (intramolecular H-bonding)], 1700 cm⁻¹ [ν (C=O)(thiazolidinone ring)], 1670 cm⁻¹ [ν (C=O)(carboxylic)], 1575 cm⁻¹ [ν (C-N)(thiazolidinone ring)], 1525 cm⁻¹ [ν (C-O)(phenolic)], and 835 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** (3.31 g, 10 mmol) and the mixture was then refluxed for 3–4 h. The solid products formed were suction filtered, washed with, and recrystallized from MeOH, and were then dried as mentioned above. Yield = 50–80%.

TABLE 1: Analytical, molar conductance (Λ_M) and molecular weight data of **I** 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
I	C ₁₆ H ₁₃ NO ₅ S	—	331 ^a (331.0)	—	57.80 (58.01)	3.75 (3.93)	4.10 (4.23)	9.50 (9.67)
II	MnC ₁₉ H ₂₃ NO ₈ S	6.2	453.1 ^b (479.9)	11.20 (11.44)	47.30 (47.51)	4.85 (4.79)	2.70 (2.92)	6.55 (6.67)
III	CuC ₁₇ H ₁₅ NO ₆ S	9.3	403.6 424.5 ^b	14.70 (14.96)	48.17 (48.06)	3.57 (3.53)	3.21 (3.30)	7.69 (7.54)
IV	ZnC ₁₇ H ₁₅ NO ₆ S	8.5	448.2 ^b (426.4)	15.20 (15.34)	47.70 (47.84)	3.30 (3.52)	3.10 (3.28)	7.62 (7.50)
V	FeC ₁₈ H ₁₉ NO ₇ SCl	13.8	461.5 ^b (484.5)	11.30 (11.56)	44.28 (44.58)	3.75 (3.92)	2.72 (2.89)	6.40 (6.60)
VI	MoC ₁₇ H ₁₅ NO ₈ S	7.1	462.6 ^b (488.9)	19.40 (19.62)	41.50 (41.73)	3.16 (3.07)	2.90 (2.86)	6.32 (6.54)

^amass spectral data, ^brast method data.

TABLE 2: IR, reflectance spectral data (cm⁻¹), and magnetic moments of 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.)
I	—	—	1525	835	—	—	Diamagnetic
II	1560	1348	1530	820	994	17450, 22320, 25300	5.90
III	1558	1333	1532	805	980	17230	2.02
IV	1565	1350	1535	810	985	—	Diamagnetic
V	1570	1350	1533	818	970	12700, 16100, 24900	5.95
VI	1575	1360	1534	800	980	—	Diamagnetic

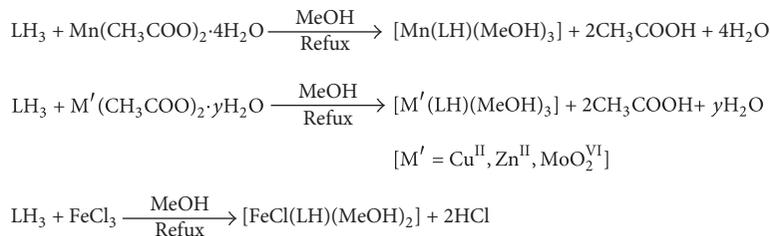
3. Results and Discussion

A dry benzene solution of the Schiff base (obtained by the nucleophilic addition reaction between 3-aldehyde-2-hydroxybenzoic acid and 2-aminophenol followed by the elimination of one water molecule) reacts with mercaptoacetic acid and forms *N*-(2-hydroxyphenyl)-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, [Mn(LH)(MeOH)₃], [M'(LH)(MeOH)] [where M' = Cu^{II}, Zn^{II}, MoO₂^{VI}] and [FeCl(LH)(MeOH)₂]. The formations of **I** from the Schiff base and the coordination compounds of the latter take place according to the Schemes 1 and 2.

The coordination compounds are stable at room temperature. They are insoluble in H₂O, partially soluble in MeOH, EtOH, and completely soluble in DMSO and DMF. Their molar conductance measurements ($\Lambda_M = 6.2 - 13.8$ 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 the Schiff base (*N*-(2-hydroxyphenyl)-3'-carboxy-2'-hydroxybenzylideneimine), LH₃ (**I**) (*N*-(2-hydroxyphenyl)-C-(3'-carboxy-2'-hydroxyphenyl)thiazolidin-4-one), and the coordination compounds of **I** 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 1630 cm⁻¹. This band disappears in **I** and a new band appears at 1575 cm⁻¹ due to the ν (C-N)(thiazolidinone ring) stretch [28] indicating the conversion of the Schiff base into **I**. The formation of **I** is further supported by the appearance of a new band at 835 cm⁻¹ due to the ν (C-S)(thiazolidinone ring) stretch [29], and it shows a negative shift by 15–35 cm⁻¹ in the coordination compounds indicating the involvement of the S atom of the thiazolidinone moiety towards coordination [30]. **I** shows the ν (C=O)(thiazolidinone ring) stretch [31] at 1700 cm⁻¹. This band remains unchanged in the coordination compounds indicating the noninvolvement of O atom towards the coordination. **I** exhibits a strong band at 2860 cm⁻¹ due to the intramolecular H-bonded [32, 33] OH group of phenolic and/or carboxylic acid moieties. 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 towards coordination. The presence of a broad band at ~3400 cm⁻¹ due to ν (O-H)(MeOH) and the decrease of ν (C-O)(MeOH) stretch from 1034 cm⁻¹ to lower energy by 40–64 cm⁻¹ in the coordination compounds of **I** indicate the involvement of the O atom of MeOH towards coordination [34]. The appearance of two new bands between 1558–1575 cm⁻¹, ν_{as} (COO) and 1333–1360 cm⁻¹, ν_s (COO) stretches indicate the presence of the coordinated carboxylate group in the coordination compounds. The energy difference

SCHEME 1: Preparative scheme of LH₃ (I).SCHEME 2: Preparative scheme of coordination compounds of LH₃ (I).

($\Delta\nu = 212 - 225 \text{ cm}^{-1}$) between these stretches is $>210 \text{ cm}^{-1}$ which indicates the monodentate nature of the carboxylate moiety [35]. The $\nu(\text{C-O})\phi$ stretch [34] of **I** occurs at 1525 cm^{-1} . This band shifts to higher energy by $5-10 \text{ cm}^{-1}$ in the coordination compounds indicating the involvement of phenolic O atom of either 3-aldehydo-2-hydroxybenzoic acid or 2-aminophenol moieties towards coordination. On the basis of steric grounds, we suggest the noninvolvement of phenolic (2-aminophenol moiety) O atom towards coordination. The absence of a band between $820-860 \text{ cm}^{-1}$ in **V** precludes the presence of the (Fe-O-Fe) bridged structure [36]. The presence of a peak at 357 cm^{-1} in **V** confirms the presence of Fe-Cl linkage [37] in the compound. **VI** exhibits the $\nu_s(\text{O}=\text{Mo}=\text{O})$ and $\nu_{as}(\text{O}=\text{Mo}=\text{O})$ stretches at 946 and 910 cm^{-1} , respectively [38]. These bands occur in the usual ranges: $\nu_s(\text{O}=\text{Mo}=\text{O})$ stretch, $892-964 \text{ cm}^{-1}$ and $\nu_{as}(\text{O}=\text{Mo}=\text{O})$ stretch, $842-928 \text{ cm}^{-1}$, reported for the majority of MoO_2^{VI} compounds [38]. The presence of two bands due to the $\nu(\text{O}=\text{Mo}=\text{O})$ stretch is indicative of a *cis*- MoO_2 configuration as the compound with *trans*- MoO_2 structure shows only $\nu_{as}(\text{O}=\text{Mo}=\text{O})$ stretch since the $\nu_s(\text{O}=\text{Mo}=\text{O})$ stretch is IR inactive [39]. The absence of a band at $\sim 775 \text{ cm}^{-1}$ in the MoO_2^{VI} compound indicates the absence of an oligomeric chain with $\cdots\text{Mo}\cdots\text{Mo}\cdots\text{Mo}\cdots$ interaction [40]. The new nonligand bands in the present coordination compounds in the low frequency region are assigned to the $\nu(\text{M-O})(550-570 \text{ cm}^{-1})$ and the $\nu(\text{M-S})(345-370 \text{ cm}^{-1})$ and these bands [41] are in the expected order of increasing energy: $\nu(\text{M-S}) < \nu(\text{M-O})$. On the basis of analytical data (Table 1), valence requirements, and the infrared spectral studies, it is proposed that **I** behaves as a dibasic tridentate OOS donor ligand in the coordination compounds.

3.2. Reflectance Spectral Studies. **II** exhibits three bands at 17450 , 22320 , and 25300 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 [42]. The presence of

an asymmetric broad band at 17230 cm^{-1} due to the ${}^2B_{1g} \rightarrow {}^2A_{1g}$, ${}^2B_{2g}$, and 2E_g transitions in **III** suggests a square-planar arrangement of **I** around Cu^{II} ion [43]. The absence of a band in the range $8000-10000 \text{ cm}^{-1}$ precludes the presence of a tetrahedral structure [44]. **V** exhibits three bands at 12700 , 16100 , and 24900 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 [45].

3.3. Magnetic Measurements. The room temperature magnetic moments of the coordination compounds of **I** are presented in Table 2. The magnetic moments of **II**, **III**, and **V** are 5.90 , 2.02 , and 5.95 B.M. , respectively. These values are indicative of magnetically dilute high-spin octahedral coordination compounds of Mn^{II} and Fe^{III} ions [46]. The **IV** and **VI** are diamagnetic as expected.

3.4. ESR Studies. The ESR spectra of **III** shows well resolved four hyperfine lines with no super hyperfine lines. The various ESR spectral parameters obtained are as follows: $g_{\parallel} = 2.26$, $g_{\perp} = 2.09$, $A_{\parallel} = 1.916 \times 10^{-2} \text{ cm}^{-1}$, $A_{\perp} = 0.467 \times 10^{-2} \text{ cm}^{-1}$, $G = 2.93$, $\alpha_{\text{Cu}}^2 = 0.87$, $(\alpha')^2 = 0.19$, $\kappa = 0.55$, and $P_d = 1.86 \times 10^{-2} \text{ cm}^{-1}$. The observed order of A and g values ($A_{\parallel} > A_{\perp}$; $g_{\parallel} > g_{\perp} > 2.0023$) indicate that the complex possesses a square planar structure [47] and the ground state [48, 49] of $\text{Cu}(\text{II})$ is predominantly $d_{x^2-y^2}$ (2B_1 as the ground state). Here g_{\parallel} and g_{\perp} values denote the effective g -values when the externally applied DC field is parallel (B_{\parallel}) and perpendicular (B_{\perp}) to the symmetry axis of the crystalline field around the paramagnetic centre, respectively. The value of geometric parameter (G), a measure of the exchange interaction, is 2.93 , and this value lies within the range [50] $2.1-3.8$, consistent with the ground state [47] $d_{x^2-y^2}$. Further the value of $g_{\parallel}/A_{\parallel}$ for the complex is 117.95 cm which lies in the range ($90-140 \text{ cm}$) as reported for square planar $\text{Cu}(\text{II})$ complexes [26]. For ionic environments, g_{\parallel} is normally ≥ 2.3 and is < 2.3 for covalent environments.

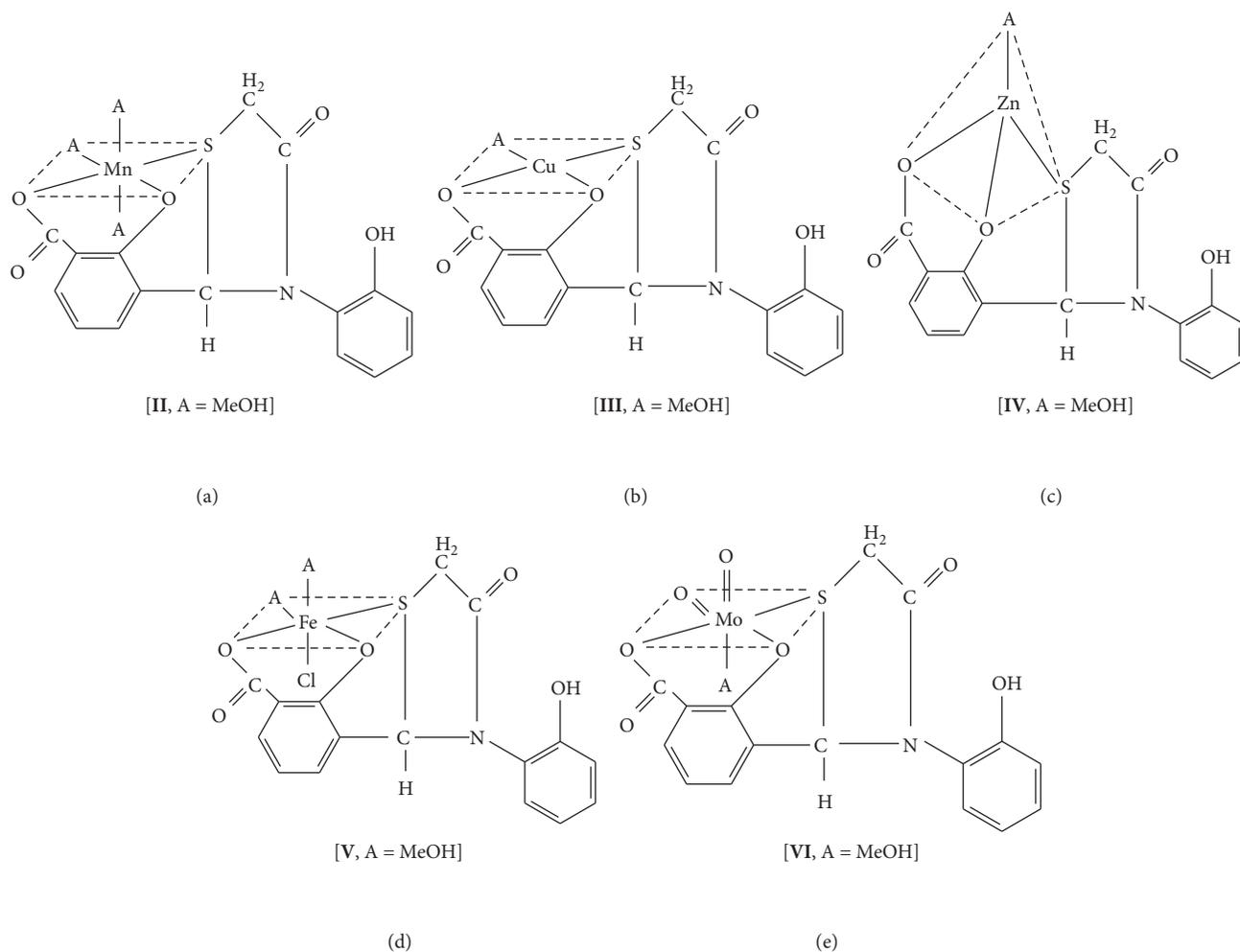


FIGURE 2

The g_{\parallel} value (2.26) indicates that the metal-ligand bonding in the compound is covalent. The G value (2.93) is less than 4.0, hence the ligand forming the Cu^{II} compound is regarded as a strong field ligand. The in-plane covalence parameter (α_{Cu}^2) has been calculated using the relation: $\alpha_{\text{Cu}}^2 = (g_{\parallel} - 2.002) + 3/7(g_{\perp} - 2.002) - (A_{\parallel}/0.036) + 0.04$, where α is related to the overlap integral (S) according to the relation [51]: $\alpha^2 - 2\alpha\alpha'S + (\alpha')^2 = 1$. The values of α_{Cu}^2 (0.87) and $(\alpha')^2$ (0.19) indicate its covalent nature. The smaller the value of α_{Cu}^2 , the more covalent is the bonding; $\alpha_{\text{Cu}}^2 = 1$ indicates completely ionic bonding, while $\alpha_{\text{Cu}}^2 = 0.5$ indicates completely covalent bonding [26]. The larger the value of $(\alpha')^2$, the more covalent is the bonding; $(\alpha')^2 = 0$ suggests a complete ionic bonding [26]. The symbol κP_d represents the Fermi contact contribution (A) to the coupling, where P_d is the dipolar contribution. The value of P_d and κ have been calculated using the relation [48, 49]: $P_d = -(A_{\parallel} - A_{\perp})/0.78$ and $\kappa = -0.48 - (A_{\parallel}/P_d)$. The positive value of κ suggests that A_{\parallel} should be greater than A_{\perp} and it has also been observed by us. The lower value of P_d in comparison to that of the free ion value ($3.5 \times 10^{-2} \text{ cm}^{-1}$) indicates the presence of covalent

character between the metal-ligand bonding. The absence of any band $\sim 1500 \text{ G}$ due to the $\Delta M_s = \pm 2$ transition rules out the presence of $M-M$ interaction.

4. Conclusion

Thus, on the basis of analytical, molecular weight, spectral, and magnetic studies, we suggest a square-planar structure, **III** for $[\text{Cu}(\text{LH})(\text{MeOH})]$, a tetrahedral structure, **IV** for $[\text{Zn}(\text{LH})(\text{MeOH})]$ and an octahedral structure, **II** for $[\text{Mn}(\text{LH})(\text{MeOH})_3]$, **V** for $[\text{FeCl}(\text{LH})(\text{MeOH})_2]$, and **VI** for $[\text{MoO}_2(\text{LH})(\text{MeOH})]$ as shown in Figure 2.

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