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Synthesis and spectroscopic study of transition metal complexes of tridentate ligand formed by direct condensation of *o*-vanillin and 2-aminophenol: X-ray structural characterization of the zinc(II) complex

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ABSTRACT

The reactions of the Schiff base 2-((2-hydroxyphenylimino)methyl)-6-methoxyphenol (H_2L), obtained by direct condensation of 2-aminophenol and 2-hydroxy-3-methoxybenzaldehyde, with some transition metal ions (Mn(II), Co(II), Ni(II), Cu(II) and Zn(II)) afforded complexes of general formulae $[M_2(L)_2(solvent)_x]$ (M: Mn, Co, Ni, Cu or Zn; Solvent: DMSO or H_2O). These compounds were characterized by elemental analysis, UV-Vis, IR, ^1H- and ^{13}C-NMR spectroscopies, molar conductivity and room temperature magnetic measurements. The structure of zinc(II) complex has been determined by X-ray crystallography. Crystal data for $C_{32}H_{34}N_2O_8S_2Zn_2$ (M = 769.47 g/mol): Orthorhombic, space group Pbca (no. 61), a = 16.3176(7) Å, b = 9.1247(3) Å, c = 21.8274(10) Å, V = 3250.0(2) Å^3, Z = 4, T = 173(2) K, μ(MoKα) = 1.658 mm^{-1}, D_{calc} = 1.573 g/cm^3, 28116 reflections measured (4.5° ≤ 2Θ ≤ 60.3°), 4457 unique (R_{int} = 0.0409, R_{sigma} = 0.0371) which were used in all calculations. The final R_1 was 0.0307(0.0466) and wR_2 was 0.0649 (0.0701) (all data). The coordination sphere of the Zn center is best described as a trigonal bipyramid.

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1. Introduction

The Schiff bases obtained by condensation of 2-amino phenol with a keto-precursor are widely used in the synthesis of transition metal complexes [1-4]. These complexes are of particular interest for the magnetism [5], catalysis [6] or medicine [7] sectors. Some of these complexes are also reported in the literature as antioxidants [8] or antibacterial agents [9,10]. Original structures are obtained thanks to the different modes of coordination of these ligands [11-14]. In recent years, these basic types of Schiff have been used to prepare metallobiosite analogues such as phenoxazinone synthase [15]. It is in this context that we decided to study the behavior of the Schiff base obtained from 2-aminophenol and *o*-vanillin in the presence of transition metals. Several complexes are isolated and characterized by different techniques.

2. Experimental

2.1. Materials and procedures

2-Aminophenol, 2-hydroxy-3-methoxybenzaldehyde as well as $M(OAc)_2{\cdot}nH_2O$ (M = Mn(II), (Co(II), Ni(II), Cu(II) or Zn(II)) were commercial products (from Alfa and Aldrich) and were used without further purification. Solvents were of reagent grade and were purified by the usual methods. Elemental analyses were performed in a Carlo-Erba EA microanalyser. Infrared spectra were recorded as KBr discs on a Bruker IFS-66V spectrophotometer. LSI-MS were recorded using a Micromass Autospec spectrometer with 3-nitrobenzyl alcohol as the matrix. The ^1H- and ^{13}C-NMR spectra were recorded in DMSO-d_6 on a Bruker 500 MHz spectrometer at room temperature using TMS as an internal reference.

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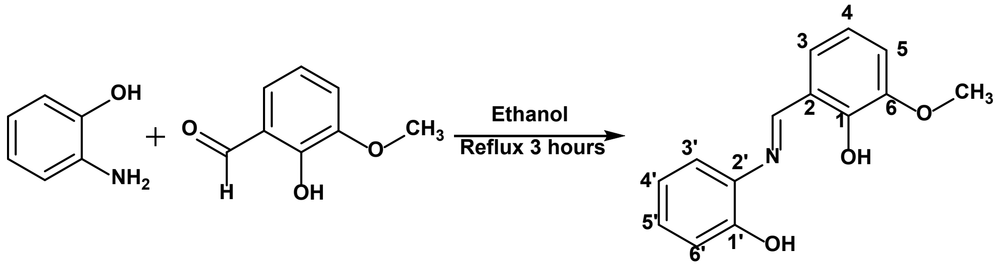
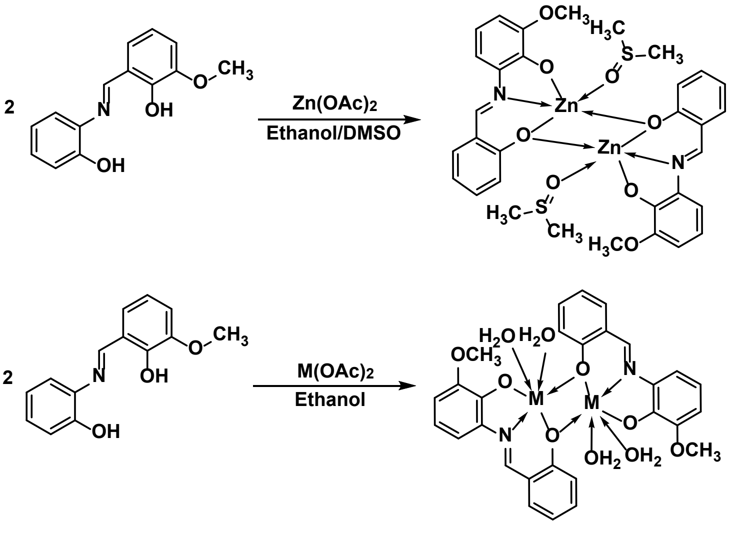


Scheme 1



Scheme 2. Proposed structures for the binuclear complexes $[M_2(L)_2(H_2O)_4]\cdot nH_2O$. M = Mn^{2+}, Cu^{2+} (n=1); M = Co^{2+}, Ni^{2+} (n=2) and $[Zn_2(L)_2(DMSO)_2]$.

The UV-Vis spectra were run on a Shimadzu UV-2501 PC Recording spectrophotometer (1000-200 nm). The molar conductance of 1×10^{-3} M in DMSO solutions of the metal complexes was measured at 25 °C using a WTW LF-330 conductivity meter with a WTW conductivity cell. Room temperature magnetic susceptibilities of the powdered samples were measured using a Johnson Mattey scientific magnetic susceptibility balance (Calibrant: $Hg[Co(SCN)_4]$). Melting points were recorded on a Büchi apparatus and are uncorrected.

2.2. Synthesis of 2-((2-hydroxyphenylimino)methyl)-6-methoxyphenol

2-Aminophenol (2 g, 21.2 mmol) and 2-hydroxy-3-methoxybenzaldehyde (3.22 g, 21.2 mmol) were refluxed during 3 hours in ethanol (40 mL). On cooling an orange precipitate appeared and was isolated by filtration. The solid was washed with cold ethanol and dried on P_2O_5 in a desiccator to yield monohydrated 2-((2-hydroxyphenylimino)methyl)-6-methoxyphenol (Scheme 1).

2-((2-Hydroxyphenylimino)methyl)-6-methoxyphenol: Color: Orange. Yield: 76%. M.p.: 202-205 °C. IR (KBr, ν, cm^{-1}): 1615 (C=N), 1599-1457 (C=C aromatic), 1328 (C-O$_{\text{phenolic}}$), 1240 (C-O$_{\text{ether}}$), 3100 (O-H (phenol) and H_2O). ^1H NMR (500 MHz, DMSO-*d*$_6$, δ, ppm): 3.80 (s, 3H, OCH_3), 6.75-7.40 (m, 7H, Ar-H), 9.00 (s, 1H, H-C=N), 9.80-10.00 (s, 2H, OH). ^{13}C NMR (125 MHz, DMSO-*d*$_6$, δ, ppm): 56.50 (OCH_3), 115 (H-C6'), 117 (H-C5), 119 (H-C4), 120 (H-C4'), 121 (H-C3), 124 (H-C3'), 128 (H-C5'), 135 (C2), 148 (C2'), 151 (C1), 153 (C1'), 153 (C6), 162 (H-C=N). UV-Vis (Liquid, λ, nm): 280, 350. MS (ESI-TOF, *m/z*): 262.1 $[H_2LH]^+$. Anal. calcd. for $C_{14}H_{15}NO_4$: C, 64.36; H, 5.79; N, 5.36. Found: C, 64.26; H, 5.81; N, 5.40%.

2.3. Synthesis of the complexes

$H_2L\cdot H_2O$ (10 mg, 0.41 mmol) was dissolved in ethanol (10 mL) to give an orange solution. The appropriate acetate salt (0.41 mmol) was added. The resulting mixture was stirred and refluxed for two hours. On cooling the suspension was filtered and the resulting solid was dried on P_2O_5 in a desiccator. A DMSO solution of the zinc(II) complex afforded by crystals suitable for X-ray analysis after one week (Scheme 2).

Tetraaqua-*bis*[μ_2-*N*-(2-oxyphenyl)-3-methoxysalicylaldiminato]-di-manganese hydrate, $[Mn_2(C_{14}H_{11}NO_3)_2(H_2O)_4].H_2O$: Yield: 58 %. IR (KBr, ν, cm^{-1}): 1605, 1478, 1388, 1307, 1229. Anal. calcd. for $Mn_2C_{28}H_{32}N_2O_{11}$: C, 49.28; H, 4.73; N, 4.10. Found: C, 49.22; H, 4.80; N, 4.08%. UV-Vis (Liquid, λ, nm): 266, 313, 400, 427, 457. μeff = 7.87 MB. Λ (Ω^{-1}.cm^2.mol^{-1}): fresh solution: 5.0; after 15 days: 12.0.

Tetraaqua-*bis*[μ_2-*N*-(2-oxyphenyl)-3-methoxysalicylaldiminato]-di-cobalt dehydrate, $[Co_2(C_{14}H_{11}NO_3)_2(H_2O)_4].2H_2O$: Yield: 58 %. IR (KBr, ν, cm^{-1}): 1606, 1480, 1381, 1307, 1296, 1229. Anal. calcd. for $Co_2C_{28}H_{34}N_2O_{12}$: C, 47.47; H, 4.84; N, 3.95. Found: C, 47.43; H, 4.80; N, 3.97%. UV-Vis (Liquid, λ, nm): 200, 266, 442. μeff = 7.02 MB. Λ (Ω^{-1}.cm^2.mol^{-1}): fresh solution: 9.0; after 15 days: 15.0.

Tetraaqua-*bis*[μ_2-*N*-(2-oxyphenyl)-3-methoxysalicylaldiminato]-di-nickel dehydrate, $[Ni_2(C_{14}H_{11}NO_3)_2(H_2O)_4].2H_2O$: Yield: 58 %. Anal. calcd. for $Ni_2C_{28}H_{34}N_2O_{12}$: C, 47.50; H, 4.84; N, 3.96. Found: C, 47.46; H, 4.86; N, 3.92%. IR (KBr, ν, cm^{-1}): 1608, 1584, 1540, 1477, 1457, 1432, 1379, 1314, 1278, 1218,

Table 1. Crystallographic data and refinement parameters for $[Zn_2(L)_2(DMSO)_2]$.

Parameters	Complex
Chemical formula	$C_{32}H_{34}N_2O_8S_2Zn_2$
Formula weight (g/mol)	769.47
Temperature (K)	173(2)
Crystal system	Orthorhombic
Space group	*Pbca*
Wavelength	0.71073 Å
Crystal size (mm)	0.090 × 0.090 × 0.110
a (Å)	16.3176(7)
b (Å)	9.1247(3)
c (Å)	21.8274(10)
Volume ($Å^3$)	3250.0(2)
Z	4
D_{calc} (g/cm^3)	1.573
µ (mm^{-1})	1.658
F(000)	1584
2θ range for data collection (°)	2.25 to 30.15
Index ranges	$-17 \le h \le 21$, $-12 \le k \le 9$, $-30 \le l \le 22$
Reflections collected	28116
Independent reflections	4457 [R(int) = 0.0409]
Data / restraints / parameters	4457 / 0 / 211
Goodness-of-fit on F^2	1.016
Final R Indices [$I>2\sigma(I)$]	$R_1 = 0.0307$; $wR_2 = 0.0649$
Final R Indices (all data)	$R_1 = 0.0466$; $wR_2 = 0.0701$
Largest diff. peak and hole (e.Å^{-3})	0.352 / -0.354

1178. UV-Vis (Liquid, λ, nm): 266, 300, 336, 433. µeff = 3.74 MB. Λ ($\Omega^{-1}.cm^2.mol^{-1}$): fresh solution: 11.0; after 15 days: 11.0.

Tetraaqua-*bis*[*µ*$_2$-*N*-(2-oxyphenyl)-3-methoxysalicylaldiminato]-di-copper hydrate, $[Cu_2(C_{14}H_{11}NO_3)_2(H_2O)_4].H_2O$: Yield: 58 %. IR (KBr, ν, cm^{-1}): 1610, 1600, 1580, 1541, 1479, 1461, 1435, 1381, 1317, 1289, 1270, 1246, 1183. Anal. calcd. for $Cu_2C_{28}H_{32}N_2O_{11}$: C, 48.07; H, 4.61; N, 4.00. Found: C, 48.05; H, 4.58; N, 3.98%. UV-vis (liquid, λ (nm)): 287, 315, 404, 435, 462. µeff = 0.64 MB. Λ ($\Omega^{-1}.cm^2.mol^{-1}$): fresh solution: 4.0; after 15 days: 6.0.

Bis[*µ*$_2$-*N*-(2-oxyphenyl)-3-methoxysalicylaldiminato]-*bis*(dimethylsulfoxide)-di-zinc, $[Zn_2(C_{14}H_{11}NO_3)_2(C_2H_6SO)_2]$: Yield: 58 %. IR (KBr, ν, cm^{-1}): 1601, 1581, 1535, 1474, 1433, 1385, 1290, 1258, 1214, 1179. Anal. calcd. for $Zn_2C_{32}H_{34}N_2S_2O_8$: C, 49.95; H, 4.45; N, 3.64. Found: C, 49.92; H, 4.42; N, 3.61%. UV-Vis (liquid, λ (nm)): 283, 342, 432. µeff = Diamagnetic. Λ ($\Omega^{-1}.cm^2.mol^{-1}$): fresh solution: 6.0; after 15 days: 8.0.

2.4. X-ray data collection, structure determination and refinement

Single crystals of $C_{32}H_{34}N_2O_8S_2Zn_2$ were grown by slow evaporation of DMSO solution of the complex. A suitable crystal was selected and mounted on a Bruker APEX-II CCD diffractometer with graphite monochromatized MoKα radiation (λ = 0.71073 Å). The crystal was kept at 173(2) K during data collection. Details of the X-ray crystal structure solution and refinement are given in Table 1. Using Olex2 [16], the structure was solved with the ShelXT [17] structure solution program using direct methods and refined with the ShelXL [18] refinement package using CGLS minimization.

3. Results and discussion

3.1. Formation of $[M_2(L)_2(Solvent)_x]\cdot yH_2O$

Formation of the title M(II) complex was readily achieved by equimolecular reaction of $M(OAc)_2.4H_2O$ (M = Mn, Co, Ni, Cu and Zn) and the Schiff base H_2L in ethanol under reflux (Scheme 2).

The instant color change of the H_2L solution upon addition of the ethanolic solution of $M(OAc)_2.4H_2O$ indicated immediate occurrence of the coordination of the Metal(II) to the metal-binding sites of the acyclic ligand. The precipitate which appears during stirring and refluxing was filtered after cooling the solution. Additional compound was recovered by slow evaporation of the filtrate. Slow evaporation of a DMSO solution of the zinc(II) compound gave crystals suitable for X-ray analysis.

3.2. Characterization

The acyclic Schiff bases H_2L have been prepared following a method well known in the literature [19,20]. The synthesis of the ligand was achieved in a one-step procedure using the direct condensation of 2-aminophenol and 3-hydroxy-2-metoxybenzaldehyde in a quantitative yield (Scheme 1). The IR spectrum of the ligand shows a moderate-intensity absorption at *ca*. 1615 cm^{-1} which is attributable to the ν(C=N). The bands in the region 1599-1457 cm^{-1} are assigned to the aromatic ν(C=C) vibration and the intense absorption near 3100-3200 cm^{-1} is assigned to ν(O-H) of the phenol group and H_2O. The ^1H and ^{13}C NMR spectra of the ligand were recorded in DMSO-*d*$_6$. The ^1H NMR spectrum of the compound revealed a singlet signal at δ 3.80 ppm which is assigned to the methoxy group (OCH_3). The complex signals appearing in the range δ 6.75-7.40 ppm are attributable to aromatic protons (Ar-H). The singlet at δ 9.00 ppm is due to a unique iminic proton (H-C=N). On the ^1H spectrum, two broad singlets signals appear in the range δ 9.8-10.0 ppm and are attributable to two phenolic protons which are implicated in hydrogen bond interaction. These observations confirm that the reaction is a condensation between the 2-aminophenol and 2-hydroxy-3-methoxybenzaldehyde. The ^{13}C NMR confirmed these facts. The signal at δ 163 ppm is assigned to –C=N group. The aromatic quaternary carbon atoms show signals at δ 148 (=*C*-OCH$_3$), 151 (=*C*-OH) and 153 ppm (=*C*-OCH$_3$) while the secondary carbon atoms signals appear at 119 (HC-*C*=) and 135 (=*C*-N). The signal of the methoxy group (O*C*H$_3$) is pointed at δ 56.50 ppm.

The reactions of H_2L with acetate metal salts in 1:1 molar ratio were investigated and the complexation was performed by mixing ethanol solutions of both ligand and metal salt. In all cases the complexes appear to be air stable and soluble in common organic solvents. Crystals suitable for X-ray analysis were obtained by slow evaporation of the DMSO solution of the zinc complex. All compounds are characterized by elemental analysis (C, N, H), IR spectroscopy, molar conductivity, magnetic measurements, and X-ray diffraction for the zinc complex. Upon complexation of H_2L with M(II) ions (M: Mn, Co,

Table 2. Electronic data, magnetic moments and conductance data of the complexes.

Compound	**UV Bands**		**Λ ($\Omega^{-1}cm^2mol^{-1}$)**		**μ/μ_B**
	π → π*, n→ π*, MLCT *d*→*d*		**Fresh**	**Two weeks after**	
$H_2L = C_{14}H_{13}NO_3$	275, 350, 460				
$[Ni_2(L)_2(H_2O)_4]\cdot 2H_2O$	266, 300, 336	433, 677	11.0	11.0	3.74
$[Cu_2(L)_2(H_2O)_4]\cdot H_2O$	287, 315, 328, 406	462, 636	4.0	6.0	0.64
$[Mn_2(L)_2(H_2O)_4]\cdot H_2O$	266, 313, 400	446, 600	5.0	12.0	5.87
$[Co_2(L)_2(H_2O)_4]\cdot 2H_2O$	200, 266, 442	470, 675	9.0	15.0	7.02
$[Zn_2(L)_2(C_2H_6SO)_2]$	283, 342, 432	-	6.0	8.0	Dia.

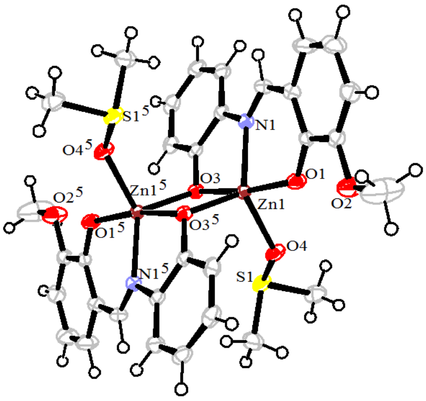


Figure 1. Crystal structure of the binuclear complex $[Zn_2(L)_2(DMSO)_2]$, showing partially the atom numbering scheme. Symmetry code (5): -*x*, -*y*, -*z*.

Ni, Cu or Zn), an significant shift of the C=N band to lower frequency is observed on comparison to the corresponding band of the free ligand. This fact is indicative of the participation of the imine group in the coordination [21].

The bands of the C-O phenolic group, appearing in the range 1240-1227 cm^{-1} in the IR spectra of the complexes are suggesting an interaction between the metal ion and the oxygen atom. Two bands in the high frequencies at ~3448 and ~3200 cm^{-1} are observed and are attributable, respectively, to the ν(OH) of the coordinated water molecule and the lattice water molecule.

The electronic spectral data of the complexes are recorded and the main bands are listed in Table 2. The assignments are made by comparison with literature data [21,22]. In the case of phenolic ligand, a π → π* band which is observed around 200-250 nm is correlated to the electrons transfer in phenyl rings [22]. The band in the regions 300-350 nm and 350-400 nm are respectively due to π → π* and n → π* electron transfer in imine function [22]. The intense band observed in the spectra of the Cu, Ni and Co complexes near 250 nm and is assigned to π → π* transition of the aromatic rings. In the region 300-445 nm intense bands are observed and are assigned to n → π* transition in imine function [21,22].

In the electronic spectra of the complexes bands of *d*→*d* transitions are observed. The electronic spectrum of the Mn(II) complex displayed two bands with very low intensities in the visible at 446 and 600 nm which are probably due, respectively, to the electronic transition $^6A_1 \rightarrow {}^4T_{2g}$ and $^6A_1 \rightarrow {}^4T_{1g}$ are in accordance with an octahedral geometry around Mn(II) ion [23]. Two bands are pointed at 433 and 677 nm in the Ni(II) complex spectrum. The third transition band has not been pointed due to the low intensity. These values are typical of octahedral Ni(II) complexes and they are attributed, respectively, to $^3A_{2g} \rightarrow {}^3T_{2g}(P)$ and $^3A_{2g} \rightarrow {}^3T_{1g}(F)$ transitions [24]. In the spectrum of the Cu (II) complex, the bands at 462 and 636 nm are both assigned to *d* → *d* transition [25, 26]. The broadness of the band at 636 nm is indicative of a hexacoordinated environment around the copper ions. For the cobalt(II) complex the bands pointed at 470 and 675 nm are assignable, respectively, to $^2E_g \rightarrow {}^2T_{2g}$ and $^2E_g \rightarrow {}^2T_{1g}(P)$ transitions. These bands are typical for an octahedral environment around Co(II) ions [27]. The spectrum of the Zn^{2+} complex which has a d^{10} metal center presents no absorption band over 400 nm.

While the Zn(II) is diamagnetic, Mn(II), Ni(II) and Cu(II) complexes are paramagnetic and exhibit room temperature magnetic moments in solid state. The magnetic moment of the manganese(II) complex is 5.87 μ_B. This value is in accordance with the presence of five unpaired electrons and is indicative of a high spin d^5 configuration. These findings support octahedral geometry around the Mn^{2+} ions [28]. The magnetic moment of the dinuclear nickel(II) complex value of 3.74 μ_B is lower than the value expected for two Ni^{2+} ions in octahedral environment. This fact is indicative of antiferromagnetic interaction between the two centers a high spin octahedral complex [29]. The value of the magnetic moment at room temperature of the dinuclear cobalt complex is 7.02 μ_B. This value is in accordance with the presence of two high-spin Co(II) ions having a spin-orbit contribution and is indicative of octahedral environments around the metal centers [30]. The dinuclear Cu^{2+} complex presents at room temperature magnetic moment of 0.64 μ_B. This value is much smaller than the expected spin only calculated value. This observation is indicative of a very strong anti-ferromagnetic coupling between the two copper(II) ions via the phenoxo oxygen atoms acting as bridges [31].

Molar conductivities were measured for freshly prepared DMF solutions and after standing for two weeks (Table 2). The conductivities increased very slightly with time in DMF for all the complexes. For both complexes, the conductance values are observed in the range 04-15 cm^2.Ω^{-1}.mol^{-1}. These observations are indicative of a non-electrolyte solution in nature [32].

3.3. Structure determination

The crystal structure of the $[Zn_2(L)_2(DMSO)_2]$ (Figure 1) was solved and refined using the Bruker SHELXTL Software Package. The complex crystallizes in the orthorhombic crystal system with the centrosymmetric space group *Pbca*. Selected bond distances and angles are listed in Table 3.

Table 3. Selected geometric parameters (Å, °).

Bond lengths			
Zn1-O1	1.9679(11)	Zn1-O3	2.0104(11)
Zn1-O4	2.0192(11)	Zn1-N1	2.0534(13)
Zn1-O3	2.1112(11)		
Bond angles			
O1-Zn1-O3	105.92(5)	O3-Zn1-O4	104.92(5)
O1- Zn1-O4	90.27(5)	O3-Zn1-N1	116.76(5)
O1- Zn1-N1	90.71(5)	O3-Zn1-O3	82.95(5)
O1-Zn1-O3	168.86(5)	O4-Zn1-N1	136.23 (5)
N1-Zn1-O3	79.12(5)	O4-Zn1-O3	93.95 (4)

The asymmetric unit consists of a dimeric complex where each of the two zinc atoms is coordinated by one ligand molecule in a tridentate fashion and one DMSO molecule. One of the phenolic oxygen atom of each ligand acts as bridge between the two zinc atoms resulting in a pentacoordinated zinc (II) ions in NO_4 inner. The ligand is coordinated to the metal ion via one azomethine nitrogen atom and two phenolic oxygen atoms. The dimethyl sulfoxide molecule solvent acts as unidentate via the oxygen atom. The $ZnNO_4$ chromophores are best described as distorted trigonal bipyramids. In fact the Addison parameter [33] $\tau = (\beta - \alpha)/60$, where α and β are the largest angles around the metal center, is a structural index which is used to classified a range of environments between the perfect square planar geometry ($\tau = 0$) and the perfect trigonal bipyramid ($\tau = 1$). The value of $\tau = 0.5438$ is indicative of a highly distorted trigonal bipyramid environment the zinc atoms. For each Zn center, the equatorial plane is occupied by two bridged phenoxo oxygen atoms and one unidentate phenoxo oxygen atom, the apical position being occupied by an azomethine nitrogen atom and an oxygen atom of a coordinated DMSO solvent molecule. The bonds lengths Zn-N is 2.0534(13) Å while the distances between the zinc atom and the phenolic oxygen atoms acting as bridges are 2.1112(11) and 2.0104(11) Å, respectively. The Zn–N distances are in the order of those found for the trigonal bipyramidal complex $[Zn_2(HL)_4](ClO_4)_4$ (HL is (2-(-(3-(dimethylamino) propylimino)methyl)-4-bromophenol)) [34]. The Zn–O bond lengths where the oxygen atom is the unidentate phenolate atom, [Zn–O, 1.9679(11) Å] are shorter than those found for Zn–O lengths where the oxygen atom acts as bridge between the two zinc ions, [Zn–O, 2.1112(11) Å] (Table 3). These values are comparable to those found for a similar complex [35, 36]. The coordination distance to the oxygen atom of the DMSO solvent, [Zn1–O4, 2.0192(11) Å] are comparable with the values found for the similar zinc trigonal bipyramidal complex [37].

4. Conclusion

The nuclearity of the complexes and the octahedral environment around Mn(II), Co(II), Ni(II) and Cu(II) are supported by the spectral data while the trigonal bipyramidal environment in the Zn(II) complex is demonstrated by the X-ray diffraction structure. In both complexes, the ligand acts as tridentate through one azomethine and two deprotonated phenolic oxygen atoms. The analytical data are in accordance with the proposed structures. The magnetic moments of the diamagnetic complexes are in accordance with the binuclear formulation. The zinc(II) complex differs from other reported complexes by its coordination number although the ligand acts similarly in all complexes. This difference is confirmed by the X-ray structure of the Zn(II) complex.

Supplementary material

CCDC-1846486 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/structures/, or by e-mailing data_request@ccdc.cam.ac.uk, or by contacting the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033

Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.
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