# Synthesis and Crystal Structures of Two New Schiff Base Cobalt(II) and Nickel(II) Complexes<sup>1</sup>

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**Abstract**—The Schiff base ligand (HL) obtained from phenylmethanamine and 5-methoxysalicylaldehyde are used as ligands for Co(II) and Ni(II) resulting in complexes  $[Co(L)_2]$  (I) and  $[Ni(L)_2]$  (II), and their solid state structures were determined by X-ray crystallography. In both complexes, weak interactions play an important role in the molecular self-assembly. Complex I was stacked up to the 2D layers by C–H···O hydrogen bonds and C–H··· $\pi$  interactions. In contrast, complex II was extended into 2D sheet by C–H···O hydrogen bonds, the C–H··· $\pi$  interactions, and edge-to-face interactions.

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## **INTRODUCTION**

The current interest in the transition metal complexes of Schiff base ligands is rapidly expanding because of their applications in biological activities [1, 2], antifertility and enzymatic activity [3], catalysis [4, 5], dyes [6], and herbicidal applications [7], as well as their intriguing variety of topologies and architectures [8-11]. It is well known that biological function of cobalt is its involvement in the coenzyme related to vitamin  $B_{12}$  [12–15] and nickel is an essential element of life by being present in a number of enzymes [16, 17]. Moreover, architectural control of molecular self-organization is of great importance for the development of functional materials [18–20]. In the crystal engineering, supramolecular metal-organic architectures assembled through coordination bonds as well as other weak cooperative interactions, such as H-bonding,  $\pi - \pi$  interactions and C-H··· $\pi$  interactions. Among these intermolecular weak forces, the H-bonding is arguably the most powerful organizing element in the design of supramolecular systems [21, 22]. The  $\pi - \pi$ interactions are important noncovalent intermolecular forces which can contribute to self-assembly or molecular recognition processes when extended structures are formed from building blocks with aromatic moieties [23–25]. The C–H $\cdots\pi$  interactions are also important noncovalent intermolecular forces similar to  $\pi - \pi$  interactions. In addition, Schiff base ligands which usually contain O,N-donor atoms have played an important role in coordination chemistry, such as may act as a bidentate N,O- [26, 27] and tridentate

bonding synthon. Potential hydrogen-bond acceptors are common in supramolecular systems. They are either an increase in dimensionality or the modi-

fication of the polymer without changing its dimensionality in coordination polymers [31]. Second, it contains two aromatic rings could form  $\pi - \pi$  interactions and C-H··· $\pi$  interactions. Herein, the cobalt(II) and nickel(II) complexes of the Schiff base ligand HL, [Co(L)<sub>2</sub>] (I) and [Ni(L)<sub>2</sub>] (II), were reported.

N,O,O-donor ligand [28], and so on, to yield multi-

nickel(II) complexes, in this paper, the Schiff-base

ligand HL was prepared by the reaction of phenyl-

methanamine with 5-methoxysalicylaldehyde. The

ligand HL has two significant features. First, the O at-

oms of 5-methoxysalicylaldehyde is a hydrogen-

In order to obtain novel Schiff base cobalt(II) and

dimensional complexes [29, 30].

## **EXPERIMENTAL**

Materials and measurements. 5-Methoxysalicylaldehyde and phenylmethanamine were purchased from Aldrich and used without further purication. Elemental analysis for C, H, and N was carried out on a PerkinElmer 2400 analyzer. X-ray crystallography was carried out using a Bruker SMART APEX II CCD diffractometer.

Synthesis of complex I. 5-Methoxysalicylaldehyde (0.30 g, 2 mmol) and phenylmethanamine (0.21 g, 2 mmol) were dissolved in an aqueous methanol solution (15 mL) and stirred 1 h to give an orange solution, which was added to a methanol solution (15 mL) of  $Co(NO_3)_2 \cdot 6H_2O$  (0.29 g, 1 mmol). The mixture was stirred for another 30 min at room temperature to give

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	Value		
Parameter	Ι	II	
Molecular weight	539.47	539.25	
Crystal system	Monoclinic	Monoclinic	
Space group	C2/c	$P2_1/c$	
Crystal size, mm	$0.27 \times 0.23 \times 0.20$	$0.26 \times 0.21 \times 0.20$	
a, Å	27.394(3)	13.5152(6)	
b, Å	5.6080(6)	10.9051(5)	
<i>c</i> , Å	18.603(2)	18.3759(9)	
β, deg	116.107(2)	111.58	
<i>Т</i> , К	298(2)	298(2)	
V, Å <sup>3</sup>	2566.3(5)	2518.6(2)	
Ζ	4	4	
$\rho_{calcd}, gm^{-3}$	1.396	1.422	
<i>F</i> (000)	1124	1128	
$\mu(MoK_{\alpha}), mm^{-1}$	0.708	0.810	
Scan mode	Multiscan	Multiscan	
Index ranges $h, k, l$	$-33 \le h \le 33, -6 \le k \le 6, -22 \le l \le 22$	$-16 \le h \le 16, -13 \le k \le 13, -22 \le l \le 22$	
Reflections collected	2449	4964	
Independent reflections $(R_{int})$	12348 (0.0464)	25904 (0.0399)	
Observed data, $I > 2\sigma(I)$	2164	4224	
Parameters	169	336	
Goodness-of-fit on $F^2$	1.030	1.006	
Final <i>R</i> indices $(I > 2\sigma(I))$	$R_1 = 0.0800$	$R_1 = 0.0400$	
R indices (all data)	$wR_2 = 0.1736$	$wR_2 = 0.0965$	
$\Delta \rho_{\min} / \Delta \rho_{\max}$ , $e  \text{\AA}^{-3}$	0.558, -0.444	0.354, -0.210	

Table 1. Crystallographic parameters and summary of data collection for structures I and II

a celadon solution and then filtered. The filtrate was kept in air for a week, forming red block crystals. The crystals were isolated, washed three times with distilled water and dried in a vacuum desiccator containing anhydrous  $CaCl_2$ . The yield was 75%.

For $C_{30}H_{28}N_2O_4Co$				
anal. calcd., %:	C, 66.79;	Н, 5.23;	N, 5.19.	
Found, %:	C, 66.62;	Н, 5.34;	N, 5.10.	

Synthesis of complex II. 5-Methoxysalicylaldehyde (0.30 g, 2 mmol) and phenylmethanamine (0.21 g, 2 mmol) were dissolved in an aqueous methanol solution (15 mL). The mixture was stirred for 1 h to give an orange solution, which was added to a methanol solution (15 mL) of Ni(NO<sub>3</sub>)<sub>2</sub> ·  $6H_2O$  (0.29 g, 1 mmol).

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The mixture was stirred for another 30 min at room temperature to give a celadon solution and then filtered. The filtrate was kept in air for a week, forming black block crystals. The crystals were isolated, washed three times with distilled water and dried in a vacuum desiccator containing anhydrous CaCl<sub>2</sub>. The yield was 68%.

For C <sub>30</sub> H <sub>28</sub> N <sub>2</sub> O <sub>4</sub>	Ni		
anal. calcd., %:	C, 66.82;	Н, 5.23;	N, 5.19.
Found, %:	C, 66.70;	Н, 5.14;	N, 5.10.

X-ray structure determinations [32]. For complexes I and II, the X-ray crystallographic data were collected on a Bruker SMART Apex II CCD diffractometer using graphite-monochromated Mo $K_{\alpha}$  ( $\lambda = 0.71073$  Å)

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Bond	$d, \mathrm{\AA}$	Bond <i>d</i> , Å		$\Delta^{**}$
-	I		II	
Co(1)–O(1)	1.922(3)	Ni(1)-O(1)	1.8333(14)	0.0887
Co(1)–N(1)	2.003(4)	Ni(1)–O(3)	1.8292(15)	0.0928
N(1)–C(7)	1.296(6)	Ni(1)–N(1)	1.9214(16)	0.0816
N(1)-C(9)	1.451(6)	Ni(1)-N(2)	1.9227(16)	0.0803
		N(1)–C(7)	1.300(2)	-0.004
		N(1)-C(9)	1.475(2)	-0.024
Angle	ω, deg	Angle	ω, deg	$\Delta^{**}$
	I		II	
O(1)Co(1)N(1)	93.70(14)	O(3)Ni(1)N(2)	93.38(6)	0.32
O(1)Co(1)N(1) <sup>i</sup>	123.80(14)	O(1)Ni(1)N(1)	93.45(6)	0.25
O(1)Co(1)O(1) <sup>i</sup>	110.188(15)	O(3)Ni(1)N(1)	86.32(6)	37.48
N(1)Co(1)N(1) <sup>i</sup>	114.304(15)	O(1)Ni(1)N(2)	87.05(6)	36.75
		O(3)Ni(1)O(1)	176.392(7)	-66.204
		N(2)Ni(1)N(1)	176.601(8)	-62.297

Table 2. Selected bond distances (Å) and angles (deg) for  $I^*$  and II

\* Symmetry code:  ${}^{i}2 - x, y, 1.5 - z$ .

\*\*  $\Delta$  is the difference of the values between complexes I and II.

radiation. The collected data were reduced using the SAINT program, and empirical absorption corrections were performed using the SADABS program. The structures were solved by direct methods and refined against  $F^2$  by full-matrix least-squares methods using the SHELXTL, version 5.1. All of the non-hydrogen atoms were refined anisotropically. All other hydrogen atoms were placed in geometrically ideal positions and constrained to ride on their parent atoms. The crystallographic data for compounds I and II are summarized in Table 1. Selected bond lengths and angles are given in Table 2.

Supplementary material for complexes I and II has been deposited with the Cambridge Crystallographic Data Centre (nos. 830984 (I) and 830985

Table 3. Geometric parameters of hydrogen bonds for I and II\*

				-
Contact D. HA	Distance, Å		Angle	
Contact D=11A	D-H	Н…А	D····A	DHA, deg
		Ι		
$C(12)-H(12)\cdots O(2)^{i}$	0.93	2.66	3.593(3)	177
		II	I	
$C(6)^{ii}-H(6)\cdots O(3)$	0.93	2.72	3.596(3)	157
* Symmetry codes: <sup>i</sup> 2 –	r v 15	-7 for I	ii 1 - r 0	5 + v + 15 - z

\* Symmetry codes:  $^{1}2 - x$ , y, 1.5 - z for I;  $^{11}1 - x$ , 0.5 + y, 1.5 - z for II.

(II); deposit@ccdc.cam.ac.uk or http://www.ccdc. cam.ac.uk).

#### **RESULTS AND DISCUSSION**

The Schiff base ligand HL in this paper was prepared by reaction of phenylmethanamine with 5-methoxysalicylaldehyde, in 83% yield in an absolute MeOH solution. Ligand HL is the yellow crystallite, stable compound in air at room temperature. It is easily soluble in common polar organic solvents, such as MeCN, MeOH, and EtOH, etc. Complexes I and II were obtained from reaction of the Schiff base ligand HL with Co(NO<sub>3</sub>)<sub>2</sub> · 6H<sub>2</sub>O and Ni(NO<sub>3</sub>)<sub>2</sub> · 6H<sub>2</sub>O in methanol, respectively. The elemental analysis is in good agreement with the chemical formula proposed for complexes I and II.

In I, the Co(II) atom is four-coordinated by two N atoms and two O atoms from two bidentate ligands HL in the usual *trans* arrangement (Fig. 1a). Analogous tetrahedral Co(II) species were previously reported in [33–37]. The bond distances of Co(1)–O(1) and Co(1)–N(1) are 1.922(3) and 2.003(4) Å, respectively, which are significantly longer than those observed in other complexes with bidentate Schiff-base ligands (1.826(2) and 1.926(2) Å) [33] and comparable to those in the similar Co(II) coordination modes (1.892(2)–1.935(2) and 1.963(2)–2.009(2) Å)



**Fig. 1.** Molecular structure of complex I (symmetry code: (i) 2 - x, y, 1.5 - z) (a); hydrogen bonds (dash lines) between the molecular structures of complex I (symmetry codes: (i) -0.5 + x, 0.5 - y, -0.5 + z; (ii) 1.5 - x, 0.5 - y, 1 - z; (iii) 2 - x, y, 1.5 - z) (b); a view of C–H… $\pi$  interactions (dash lines) in I built up by symmetry operations 2 - x, y, 1.5 - z; 2 - x, -1 + y, 1.5 - z; x, -1 + y, z (c) and a view of a two-dimensional sheet of I formed by interaction of the C–H…O hydrogen bonds and C–H… $\pi$  interactions. Hydrogen atoms were omitted for clarity (d).

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**Fig. 2.** Molecular structure of complex **II** (a); 1D zigzag chain assembled *via* C–H···O hydrogen bonds in **II**. Symmetry code: (i) 1 - x, 0.5 + y, 1.5 - z; (ii) 1 - x, -0.5 + y, 1.5 - z (b); a view of C–H··· $\pi$  interactions in **II** built up by symmetry operation 1 - x, -y, 2 - z (c); the edge-to-face interactions in the neighboring molecules of **II**. Hydrogen atoms were omitted for clarity (d); a view of a 2D sheet in **II** formed by the C–H···O hydrogen bonds interactions, C–H··· $\pi$  interactions and edge-to-face interactions. Hydrogen atoms were omitted for clarity (e).

[34–37]. The angles subtended at the  $Co^{2+}$  ion in the distorted-tetrahedral geometry ( $CoN_2O_2$ ) is in the range from 93.70(14)° to 123.80(14)°. The angle between two six-membered chelate planes was 79.6(9)°. In addition, the benzene rings of the Schiff base ligand were twisted, at an angle of 84.19° with respect to each other.

It should be noted that there are persistent onedimensional C-H···O hydrogen bond interactions between the adjacent  $[Co(L)_2]$  molecules (Fig. 1b, Table 3). Desiraju studies [38] have revealed that the C···O distance in C–H···O hydrogen bond is in the range of 3.0–4.0 Å. The C–H···O angle  $\theta$  in the range of 110°–180° is acceptable, although a linear C–H···O bond (150° <  $\theta$  < 180°) is structurally more important. In I, the C(12)···O(2)<sup>i</sup> distance of 3.593(7) Å and C(12)–H(12)···O(2)<sup>i</sup> angle of 177° indicate an effective C–H···O hydrogen bond. In fact, such long C–H···O hydrogen bonds have also been observed in the literature before [39–41]. In addition, C–H··· $\pi$  interactions are also apparent in I. C–H··· $\pi$ 



Fig. 2. (Contd.)

interactions can be depicted by the distance (D < 4.3 Å) between the donor carbon atom and the centre of the acceptor ring; the distance (d < 3.4 Å) between the H of donor carbon atom and the centre of the acceptor ring; the angle ( $\theta < 25^{\circ}$ ) between the ring normal and a vector connecting the methyl carbon atom and the centre of the ring; and the angle ( $\phi > 120^\circ$ ) between the C–H and ring centre-H vectors [42, 43]. The C(8) atom forms a close contact with an aromatic ring (the ring defined by  $C(1)\cdots C(6)$  atoms) of an adjacent molecule ( $D = 3.7688 \text{ Å}, d = 2.8631 \text{ Å}, \theta < 18^{\circ}$ ,  $\varphi = 157.667^{\circ}$ ) indicate a strong C–H··· $\pi$  interaction and the adjacent molecules were connected through intermolecular C-H··· $\pi$  interactions (Fig. 1c). Together with the C-H-O hydrogen bonds, the  $C-H\cdots\pi$  interactions link the molecules of complex I into a 2D layered structure as shown in Fig. 1d.

In contrast to I, the Ni(II) atom lies on a crystallographic inversion centre. Nickel atom is four-coordibase ligands HL, which affords a planar trans-[NiN<sub>2</sub>O<sub>2</sub>] coordination geometry (Fig. 2a). Analogous tetrahedral Ni(II) species were previously reported in [44-48]. In II, the Ni(1)–O(1) bond distance of 1.8333(14) Å is slightly longer than that of Ni(1)–O(3) 1.8292(14) Å. The Ni–O bond distances are smaller than that of 1.905(3) Å observed in other analogous complexes [44] and comparable to those in the similar Ni(II) coordination modes (1.804–1.847 Å) [45–48]. The Ni(1)–N(2) bond distance of 1.9227(16) Å is slightly longer than that of Ni(1)–N(1) 1.9214(16) Å, which is significantly smaller than those observed in other analogous complexes [44]. The angles subtended at the  $Ni^{2+}$  ion in the planar geometry ( $NiN_2O_2$ ) are in the range of  $86.32(6)^{\circ}-93.45(6)^{\circ}$  and  $176.392(7)^{\circ}-$ 176.601(8)°. It is interesting to point out that two sixmembered chelate planes were twisted by 5.74(4)°

nated by two N atom and two O atoms from two Schiff

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with respect to each other, which are significantly smaller than complex **I**.

There were several significantly weak interactions in **II**. Firstly, there was a kind of C–H…O hydrogenbonding interactions between the adjacent molecules of II (Fig. 2b). The H(6)…O(3) distance was 2.72 Å with a corresponding O…C separation of 3.596(3) Å. The angle C-H···O was 157°, leading to a description of this interaction as a linear hydrogen bond [38]. Secondly, one C(9) atom of the amine was also able to interact with the aromatic ring (the ring defined by  $C(16)\cdots C(21)$  atoms) of an adjacent molecule (D =3.3469 Å, d = 2.6786 Å,  $\theta < 18^{\circ}$ ,  $\phi = 126.416^{\circ}$ ) (Fig. 2c) [42, 43]. What's more, compared with I, the unusual feature of II is the existing edge-to-face interactions. These energetically favorable nonbonded edge-to-face interactions can be defined formally as those pairs with phenyl ring centroid separations >4.5 Å and < 7 Å and dihedral angles within 30° of 90° [49]. Here, the centroid–centroid distances were 4.687 Å (Fig. 2d), and at the lower end of the range (4.5-7.0 Å)and the dihedral angles were  $60.247(7)^{\circ}$ . Together with the C(6)<sup>i</sup>–H(6)···O(3) interactions and the C–H··· $\pi$ interactions, the edge-to-face interactions extend the molecular structures of II into the 2D sheet (Fig. 2e).

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