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# Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry

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A Novel 1-D Stacking Z-Type Supramolecular Complex, [Ni(sphs)(iz)] · EtOH (sphs=4-(N-Salicylaldehyde Schiff Base) Benzeneformylhydrazone-N'-salicylaldehyde, iz=Imidazole), Constructed by Hydrogen Bonds

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# A Novel 1-D Stacking Z-Type Supramolecular Complex, [Ni(sphs)(iz)] · EtOH (sphs = 4-(N-Salicylaldehyde Schiff Base) Benzeneformylhydrazone-N'-salicylaldehyde, iz = Imidazole), Constructed by Hydrogen Bonds

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A new ligand, 4-(N-salicylaldehyde Schiff base) benzeneformylhydrazone-N'-salicylaldehyde ( $H_3L$ ), and a new quaternary complex 1, [Ni(sphs)(iz)] · EtOH (sphs =  $H_3L$ , iz = imidazole), has been designed and synthesized. The structural unit of complex 1 consists of the following groups: one Ni(II), one molecular  $H_3L$ , one imidazole and one ethanol. It belongs to the orthorhombic system and has a Pbca space group. One deprotonated oxygen in enol-form, one deprotonated oxygen of hydroxyl and one nitrogen of metheleneimine for  $H_3L$  and one nitrogen atom of imidazole coordinated to central Ni with two oxygen and two nitrogen in anti-form, respectively, constructed distorted square geometry, an ethanol existed in crystal lattice. There are intramolecular and intermolecular hydrogen bonds in the crystal structure.

Keywords Schiff base, imidazole, aroylhydrazone, Ni(II) complex

#### INTRODUCTION

The chemistry of Schiff bases and Schiff base hydrazones has been intensively investigated in recent years due to their coordination properties and diverse applications. In particular, Schiff base hydrazones and their complexes have been studied

for their antifungal and antibacterial activity as also for their potential as antiviral drugs.<sup>[1-6]</sup> Polydentate aroylhydrazones have also been the subject of extensive investigation owing to their versatile chelating behavior for which they are widely used in analytical chemistry as a selective metal extracting agent as well as in spectroscopic determination of certain transition metals.<sup>[1]</sup> Imidazole is of considerable interest as a ligand in many biological systems in which it provides a potential binding site for metal ions.<sup>[7]</sup> Imidazole itself is an unidentate ligand and forms complexes with metal ions through its tertiary nitrogen atons.<sup>[8]</sup> The self-assembly processes of coordination compounds into well-defined architectures is of current interest in supramolecular chemistry.<sup>[9,10]</sup> The svnthetic effort is oriented towards discrete species with predetermined nuclearities and topologies of the metallic centers (molecular squares and rectangles, boxes, grids, ladders, racks, helicates)<sup>[11]</sup> and towards compounds exhibiting extended structures, 1D, 2D, 3D, with a large variety of solid-state architectures.<sup>[9,12]</sup> In continuing our previous work,<sup>[13]</sup> 4-(N-salicylaldehyde Schiff base) benzeneformylhydrazone-N'-salicylaldehyde ( $H_3L$ ) (as shown in Scheme 1) has been synthesized using 4-amino-benzenecarboxylic acid, hydrazine hydrate and salicylaldehyde as the starting materials. It is of interest that H<sub>3</sub>L contains not only Schiff base hydrazones but also the Schiff base of salicylaldehyde and is actually a pentadentate ligand. Complex 1 was obtained through the self-assembly of ligands H<sub>3</sub>L and imidazole with  $NiCl_2 \cdot 6H_2O$  in anhydrous ethanol. Usually, aroylhydrazones exist in keto-form, or in enol-form and its deprotonated conjugated system. In complex 1  $H_{3}L$  is coordinated to Ni(II) through a deprotonated oxygen atom in enol-form and a

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(c) the structure of H<sub>3</sub>L in deprotonated conjugate-form

SCH. 1. H<sub>3</sub>L.

nitrogen atom of methyleneimine and deprotonated oxygen atom of hydroxyl.

#### **EXPERIMENTAL**

#### Materials and Physical Techniques

All chemicals were of analytical reagent grade quality obtained from commercial sources and used without further purification. The IR spectra were recorded on a Nicolet IR-470 spectrometer using KBr pellets in the range of 4000–400 cm<sup>-1</sup>. The thermal decomposition experiment was carried out using NETZSCH TG 209 instrument in N<sub>2</sub> atmosphere with the heating rate of  $10^{\circ}$ C min<sup>-1</sup>. X-ray crystal diffraction was measured using a Rigaku-Raxis-IV X-ray diffractometer.

#### Synthesis of the Ligand H<sub>3</sub>L

The ligand H<sub>3</sub>L was prepared by the method described in literature,<sup>[14]</sup> and the procedure modified as shown in Scheme 2.

Synthesis of ethyl 4-amino-benzenecarboxylate: 4-aminobenzenecarboxylic acid 6.9 g (0.0503 mol) and 40 mL anhydrous ethanol were added to a 100 mL three-necked bottle. To this, 3 mL of concentrated  $H_2SO_4$  under cooling was added drop wise and stirred. The mixture was heated under reflux for 3 h with stirring. 30 mL of ethanol was evaporated out and cooled to room temperature. 40 mL of water was added to the mixture and was then neutralized to pH = 7–8 using a saturated solution of Na<sub>2</sub>CO<sub>3</sub>. The white solid was separated, filtered, washed three times using water, and dried at  $50-60^{\circ}$ C Yield: 4.7 g of ethyl 4-amino-benzenecarboxylate (60%).

Synthesis of 4-amino-benzenecarbonylhydrazine: 4.7 g (0.0285 mol) of ethyl 4-amino-benzenecarboxylate and 10 mL of hydrazine hydrate were dissolved in 30 mL of anhydrous ethanol in a 100 mL three-necked bottle while stirring. The mixture was heated under reflux for 3 h with stirring. 30 mL of ethanol and residual hydrazine hydrate was evaporated, cooled to room temperature, filtered, washed three times using water, and finally dried at  $50-60^{\circ}$ C. Yield: 3.5 g of 4-amino-benzenecarbonylhydrazine (50%).

Synthesis of 4-(N-salicylaldehyde Schiff base)benzeneformylhydrazone-N'-salicyl-aldehyde: 3.5 g (0.0232 mol) of 4-amino-benzenecarbonylhydrazine and 5.7 g (0.0464 mol) salicylaldehyde were reacted in 30 mL anhydrous ethanol, the procedure was similar to the synthesis of 4-amino-benzenecarbonylhydrazine. Yield: 3.5 g 4-(N-salicylaldehyde Schiff base)benzeneformylhydrazone-N'-salicylaldehyde(H<sub>3</sub>L) (80%). M.P. 241–243 °C. IR (KBr, cm<sup>-1</sup>): 3471 m ( $\nu$  O-H), 3252 m ( $\nu$  N-H), 2977 m ( $\nu$  C-H), 1706 s ( $\nu$  C=O), 1620 s ( $\nu$  C=N), 1572 s ( $\nu$  C=C).

#### Synthesis of the complex [Ni(sphs)(iz)] · EtOH 1

 $H_3L$  0.0359 g (0.1 mmol) and imidazole 0.0068 g (0.1 mmol) were dissolved in 20 mL of anhydrous ethanol



Structure of H3L in keto-form

SCH. 2. The prepared procedure of the ligand H<sub>3</sub>L.

TABLE 1 Crystallographic data and structural refinement parameters for the complex **1** 

Formula	C <sub>26</sub> H <sub>25</sub> N <sub>5</sub> Ni O <sub>4</sub>
Formula weight	530.22
<i>T</i> (K)	291(2) K
Crystal system	Orthorhombic
Space group	Pbca
Crystal size(nm)	$0.20 \times 0.17 \times 0.16$
<i>a</i> (Å)	20.723(4)
<i>b</i> (Å)	10.618(2)
<i>c</i> (Å)	22.653(5)
V	4984.7(17)
$D_{\text{calc.}}$ (Mg/m <sup>3</sup> )	1.413
Z	8
Reflections collected/unique	11207/3943
Data/restraints/parameters	3943/1/324
$R_1, wR_2 [I > 2\sigma(I)]$	0.0644, 0.1338

while stirring using 0.1 mol L<sup>-1</sup> NaOH solution neutralized to pH = 7. 10 mL of ethanol solution containing 0.0238 g (0.1 mmol) NiCl<sub>2</sub> · 6H<sub>2</sub>O was added to the above solution. The resulting mixture was heated under reflux for 0.5 h, cooled to ambient temperature, then allowed to stay at this temperature. After three weeks, green crystals suitable for X-ray diffraction were formed from the mother solution, filtered, washed using water, and dried in air. Yield: 0.024 g crystals of complex **1** [Ni(sphs)(iz)] · EtOH (45.3%). IR (KBr, cm<sup>-1</sup>): 3423 m ( $\nu$  O-H), 3149 m ( $\nu$  N-H <sub>iz</sub>), 2971 m ( $\nu$  C-H), 1603 s ( $\nu$  C=N), 1572 s ( $\nu$  C=C), 1540 s ( $\nu$  C=N)<sub>im</sub>.

#### **Crystal Structure Determination**

Crystal data for complex **1** were collected on a Rigaku-Raxis-IV X-ray diffractometer using graphite-monochromated Mo K $\alpha$  radiation ( $\lambda = 0.7103$  Å) at 291(2) K. 11207 Reflections were measured over the ranges  $1.80 \le 2\theta \le 25.00^\circ$ ,  $-24 \le h \le 24$ ,  $0 \le k \le 12$ ,  $-26 \le l \le 26$ , yielding 3943 unique reflections. Raw data were corrected and the structure was solved using the SHELX-97 program. Non-hydrogen atoms were located by direct phase determination and subjected to anisotropic refinement.<sup>[15]</sup> The full-matrix least squares calculations on  $F^2$  were applied on the final refinement. The refinement converged at  $R_1 = 0.0644$  and  $wR_2 = 0.1338$  values for reflections with  $I > 2\sigma$  (I). The crystallographic data and structural refinement parameters of complex **1** are listed in Table 1. Details of crystal structure determination are described next. Full atomic data are available as a file in CIF format.

#### **RESULTS AND DISCUSSION**

#### Crystal Structure Described for the Complex 1

Selected bond distances and bond angles and hydrogenbond data of complex **1** are listed in Tables 2 and 3. The crystal structural unit of complex **1** consists of one Ni(II), one molecular H<sub>3</sub>L, imidazole and ethanol. It is shown in Figure 1 that one deprotonated oxygen in enol-form, one deprotonated oxygen of hydroxyl and one nitrogen of metheleneimine for H<sub>3</sub>L and one nitrogen atom of imidazole coordinated to central Ni(1) with two oxygen and two nitrogen in anti-form, respectively, constructed distorted square geometry. The bond distances Ni(1)-O(2) and Ni(1)-N(1) are 1.857(3) Å and 1.825(4) Å, respectively, both of which are shorter than the literature values (2.026(2), 2.073(3) Å).<sup>[16]</sup>

TABLE 2Selected bond distances (Å) and angles (°) for the complex 1

		pron 1
1.821(3)	C(24)-N(4)	1.318(6)
1.825(4)	C(22)-N(4)	1.381(6)
1.853(3)	C(15)-N(3)	1.286(6)
1.909(4)	C(12)-N(3)	1.416(6)
1.332(5)	C(17)-O(3)	1.371(6)
1.313(5)	C(8)-N(2)	1.317(6)
1.393(5)	C(7)-N(1)	1.305(6)
96.23(16)	O(1)-Ni(1)-O(2)	179.52(15)
83.37(16)	O(1)-Ni(1)-N(4)	89.61(15)
173.40(18)	O(2)-Ni(1)-N(4)	90.80(15)
118.6(4)	O(1)-C(1)-C(6)	123.7(4)
124.3(5)	C(7)-N(1)-N(2)	117.4(4)
127.0(4)	N(2)-N(1)-Ni(1)	115.7(3)
108.5(4)	C(15)-N(3)-C(12)	119.6(4)
126.6(3)	C(22)-N(4)-Ni(1)	128.5(3)
126.8(3)	C(8)-O(2)-Ni(1)	111.2(3)
	$\begin{array}{c} 1.821(3) \\ 1.825(4) \\ 1.853(3) \\ 1.909(4) \\ 1.332(5) \\ 1.313(5) \\ 1.393(5) \\ 96.23(16) \\ 83.37(16) \\ 173.40(18) \\ 118.6(4) \\ 124.3(5) \\ 127.0(4) \\ 108.5(4) \\ 126.6(3) \\ 126.8(3) \end{array}$	$\begin{array}{ccccc} 1.821(3) & C(24)-N(4) \\ 1.825(4) & C(22)-N(4) \\ 1.853(3) & C(15)-N(3) \\ 1.909(4) & C(12)-N(3) \\ 1.332(5) & C(17)-O(3) \\ 1.313(5) & C(8)-N(2) \\ 1.393(5) & C(7)-N(1) \\ 96.23(16) & O(1)-Ni(1)-O(2) \\ 83.37(16) & O(1)-Ni(1)-N(4) \\ 173.40(18) & O(2)-Ni(1)-N(4) \\ 118.6(4) & O(1)-C(1)-C(6) \\ 124.3(5) & C(7)-N(1)-N(2) \\ 127.0(4) & N(2)-N(1)-Ni(1) \\ 108.5(4) & C(15)-N(3)-C(12) \\ 126.6(3) & C(22)-N(4)-Ni(1) \\ 126.8(3) & C(8)-O(2)-Ni(1) \\ \end{array}$

Hydrogen-bolids for complex I [A and ]							
D-HA	d(D-H)	d(HA)	d(DA)	<(DHA)			
O(4)-H(4)N(2)#1 N(5)-H(5B)O(4) O(3)-HN(3)	0.82 0.86	2.03 1.92	2.815(5) 2.775(5) 2.612	161.0 170.3			

 TABLE 3

 Hydrogen-bonds for complex 1 [Å and °]

Symmetry transformations used to generate equivalent atoms: #1 x, y + 1, z.

The bond distance for Ni(1)-O(1) = 1.821(3) Å is consistent with literature  $(1.816 \text{ Å});^{[15]}$  Ni(1)-N(4) = 1.909(4)Å is shorter than the literature value (2.060(4)).<sup>[17]</sup> The bond  $N(1)-Ni(1)-O(1) = 96.23(16)^{\circ}$ , N(1)-Ni(1)angles are:  $O(2) = 83.37(16)^{\circ},$  $O(1)-Ni(1)-N(4) = 89.61(15)^{\circ}, O(2) Ni(1)-N(4) = 90.80(15)^{\circ},$  $O(1)-Ni(1)-O(2) = 179.52(15)^{\circ}$ . benzene-rings (C(1)C(2)C(3)-C(4)C(5)C(6))Two and (C(8)C(9)C(10)C(11)C(12)C(13)C(14)) linked through the acylhydrazone are almost on a plane with the deviation angle of 4.5°. The deviation angle is 16.7° between the benzene-ring (C(8)C(9)C(10)C(11)C(12)C(13)C(14)) and the imidazole ring (C(21)C(22)C(23)N(4)N(5)), the deviation angle is 13° of (C(1)C(2)C(3)C(4)C(5)C(6)) and (C(21)C(22)C(23)N(4)N)(5)),  $43.5^{\circ}$  of (C(15)C(16)C(17)C(18)C(19)C(20)C(21)) and (C(9)C(10)C(11)C(12)C(13)C(14)-N(3)). An ethanol molecule as lattice solvent does not participate in coordination. There are two kinds of hydrogen bonds in the crystal structural unit (Figure 2): one is an intramolecular hydrogen bond (O(3)-H...N(3) = 2.612 Å) formed from the hydrogen atom of the hydroxyl and nitrogen atom of the methyleneimine of Schiff base, the other is an intermolecular hydrogen bond (N(5)-H... O(4) = 2.775(5) Å) of the oxygen atom of ethanol with the hydrogen atom of methyleneimine of

imidazole. The hydrogen atom of ethanol can also form a hydrogen bond [O(4)-H...N(2) = 2.815(5) Å] with the nitrogen atom of the acylhydrazone methyleneimine adjoining crystal structural unit, resulting in the crystal being extended into 1D chain (as shown in Figure 3) along the *b* axis. Because of intermolecular weak interaction (as  $\pi$ - $\pi$  interaction et al.), the crystal structural units were packed into a supramolecular compound with Z type architecture.

#### **IR Spectra**

In the IR spectrum of the ligand H<sub>3</sub>L, the absorption bands of  $\nu$  (O-H),  $\nu$  (N-H) and  $\nu$  (C-H) appeared at 3471, 3252, 2971 cm<sup>-1</sup> and the strong absorption bands of the  $\nu$  (C==O),  $\nu$  (C=N),  $\nu$  (C=C) appeared at 1706, 1620, 1572 cm<sup>-1</sup>, respectively. These results showed that H<sub>3</sub>L exists in ketoform and contains the hydroxyl from salicylaldehyde. In the IR spectrum of complex 1, the absorption bands of  $\nu$  (C=O) and  $\nu$  (N-H) disappeared, the bands of  $\nu$  (O-H) and  $\nu$  (C=N) were shifted to a lower frequency about 48 and 13 cm<sup>-1</sup> and the absorption bands of the  $\nu$  (N-H) and  $\nu$  (C=N) of imidazole were observed at 3148 and  $1540 \text{ cm}^{-1}$ . It is seen from the results that H<sub>3</sub>L in enol-form exists in complex 1, deprotonated carbonyl oxygen, hydroxyl oxygen and methyleneimine nitrogen coordinated to Ni(II), the nitrogen atom of imidazole also participated in coordination. In the IR spectra of the ligand  $H_{3}L$  and complex 1, a broad absorption band was observed at



FIG. 1. Structural unit of the complex 1 with atom numbering.



FIG. 2. Extensive structure of structure unit of the complex 1 along the *b* axis with hydrogen bonding.



FIG. 3. Packing structure of the complex 1 viewed along b axis, showing network of Z type.

the  $3500-2500 \text{ cm}^{-1}$  range, which indicates the existence of hydrogen bonding. The IR spectral result of complex **1** is consistent with the conclusion of crystal structural analysis.

#### Thermal Behavior

In TG-DTG curve of the complex **1** the first strong DTG peak was at  $114-148^{\circ}$ C range with a weight loss of 7.75% corresponding to 1 mol of ethanol lost per formula unit (calc. 8.67%). A weak DTG peak was observed between  $148-362^{\circ}$ C with the weight of loss was 12.25%, corresponding to 1 mol. of imidazole left per formula unit (calc. 12.26%). At  $362-658^{\circ}$ C range there are four small peaks, and the total weight loss was 56.11%, owing to the decomposition of the ligand HL (calc. 67.36%). The residue was 23.89%, which may be composed of the newborn NiO (14.1%) and the carbonized products of the organic compounds.

#### SUPPLEMENTARY MATERIAL

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 275416 for complex 1. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk or www: http:// www.ccdc.cam.ac.uk).

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