Synthesis, Characterization and Crystal Structures of Cobalt(II), Zinc(II) and Cadmium(II) Complexes Derived from 2-Hydroxy-*N'*-(pyridin-2-ylmethylene)benzohydrazide with Antimicrobial Activity

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Abstract

Three new cobalt(II), zinc(II) and cadmium(II) complexes, $[\text{CoL}_2] \cdot 2\text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$ (**1**), $[\text{ZnL}_2]$ (**2**) and $[\text{Cd}(\text{HL})_2(\text{NO}_3)]\text{N}_3$ (**3**), were prepared from 2-hydroxy-*N'*-(pyridin-2-ylmethylene)benzohydrazide (HL). The complexes have been characterized by IR, UV-Vis and single-crystal X-ray diffraction techniques. X-ray analysis indicates that the complexes are mononuclear species, with the metal atoms in octahedral coordination. The hydrazone compound and its complexes were evaluated for their antibacterial (*Bacillus subtilis*, *Staphylococcus aureus*, *Escherichia coli*, and *Pseudomonas fluorescence*) and antifungal (*Candida albicans* and *Aspergillus niger*) activities. The complexes have effective activities against the bacteria.

Keywords: Hydrazone; Mononuclear complex; Crystal structure; Antimicrobial activity

1. Introduction

Hydrazones are a kind of Schiff base compounds, bearing typical functional group, $\text{CH}=\text{N}-\text{NH}-\text{C}(\text{O})$. Compounds have attracted tremendous attention due to their interesting and versatile biological properties like antibacterial,¹ antifungal,² as well as antitumor.³ Cobalt, zinc and cadmium complexes derived from Schiff base ligands have been widely studied for their potential antibacterial activities.⁴ As a further exploration of novel complex based antimicrobial agents, in the present work, three new cobalt(II), zinc(II) and cadmium(II) complexes, $[\text{CoL}_2] \cdot 2\text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$ (**1**), $[\text{ZnL}_2]$ (**2**) and $[\text{Cd}(\text{HL})_2(\text{NO}_3)]\text{N}_3$ (**3**), respectively, where L is the anion of 2-hydroxy-*N'*-(pyridin-2-ylmethylene)benzohydrazide (HL; Scheme 1), are presented.

Scheme 1. HL

2. Experimental

2. 1. Materials and Instruments

All the organic compounds and inorganic salts with AR grade were obtained from Sigma-Aldrich and used without further purification. The ligand HL was prepared according to the literature method.⁵ Elemental analyses (C, H, N) were carried out with a Perkin-Elmer 2400II analyzer. FT-IR spectra were recorded on a Perkin-Elmer 377 spectrometer with KBr pellets. UV-Vis spectra were recorded on a Lambda 900 spectrometer. ¹H and ¹³C NMR data were recorded on a Bruker 500 MHz spectrometer. X-ray diffraction was carried out on a Bruker APEX II CCD diffractometer.

2. 2. Synthesis of Complex 1

HL (1.0 mmol, 0.24 g) and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (1.0 mmol, 0.29 g) were dissolved in 50 mL MeOH. The mixture was heated to reflux for 1 h. Then, it was cooled to room temperature. Single crystals of the complex were generated after 5 days following the slow evaporation of the solvent. Yield 27%. IR data (cm^{-1}): 3454 (OH), 1600 (C=N). UV-Vis data (CH_3OH , λ_{max} , nm): 276, 380, 390.

Elemental analysis found: C, 54.25; H, 4.72; N, 13.46%, $C_{26}H_{20}CoN_6O_4 \cdot 2CH_3OH \cdot H_2O$ calcd: C, 54.11; H, 4.87; N, 13.52%.

2. 3. Synthesis of Complex 2

HL (1.0 mmol, 0.24 g) and $Zn(CH_3COO)_2 \cdot 2H_2O$ (1.0 mmol, 0.22 g) were dissolved in 50 mL MeOH. The mixture was heated to reflux for 1 h. Then, it was cooled to room temperature. Single crystals of the complex were generated after 7 days following the slow evaporation of the solvent. Yield 45%. IR data (cm^{-1}): 3450 (OH), 3387 (NH), 1596 (C=N). UV-Vis data (CH_3OH , λ_{max} , nm): 269, 305, 323, 370. Elemental analysis found: C, 57.38; H, 3.77; N, 15.26%, $C_{26}H_{20}N_6O_4Zn$ calcd: C, 57.21; H, 3.69; N, 15.40%.

2. 4. Synthesis of Complex 3

HL (1.0 mmol, 0.24 g), NaN_3 (1.0 mmol, 0.065 g) and $Cd(NO_3)_2 \cdot 4H_2O$ (1.0 mmol, 0.31 g) were dissolved in 50 mL MeOH. The mixture was heated to reflux for 1 h. Then, it was cooled to room temperature. Single crystals of the complex were generated after 3 days following the slow evaporation of the solvent. Yield 33%. IR data (cm^{-1}): 3450 (OH), 2072 (N_3), 1638 (C=O), 1601 (C=N), 1456, 1311, 1072 (NO_3). UV-Vis data (CH_3OH , λ_{max} , nm): 280, 368. Elemental analysis found: C, 44.52; H, 3.31; N, 19.92%, $C_{26}H_{22}CdN_{10}O_7$ calcd: C, 44.68; H, 3.17; N, 20.04%.

2. 5. X-ray Crystallography

The collected data were reduced with SAINT,⁶ and multi-scan absorption correction was applied by using SADABS.⁷ Structures of the three complexes were solved by direct method and refined against F^2 by full-matrix least-squares method with SHELXTL.⁸ The non-hydrogen atoms were refined anisotropically. The hydroxyl and amino hydrogen atoms were located from electronic density maps and refined isotropically. The remaining hydrogen atoms were placed in calculated positions and constrained to ride on their parent atoms. The atoms O5, O6, O7, C27 and C28 of the solvent molecules in complex 1, and the azide anion in complex 2 are disordered, which were refined with isotropic behavior. The hydrogen atoms of the disordered methanol molecules in complex 1 are not added, but included in the formula. The crystallographic data and refinement parameters for the compounds are listed in Table 1. Selected bond lengths and angles are listed in Table 2.

3. Results and Discussion

3. 1. Chemistry

HL was prepared from the condensation reaction of 2-pyridinecarboxaldehyde with 2-hydroxybenzohydrazide in MeOH. The complexes were prepared from the self-assembly of hydrazone compound with cobalt nitrate, zinc acetate, and cadmium nitrate and sodium azide, respec-

Table 1. Crystallographic and refinement data for HL and its metal complexes.

	HL	1	2	3
Formula	$C_{13}H_{11}N_3O_2$	$C_{28}H_{28}CoN_6O_7$	$C_{26}H_{20}N_6O_4Zn$	$C_{26}H_{22}CdN_{10}O_7$
Formula weight	241.25	618.49	545.85	698.94
T (K)	298(2)	298(2)	298(2)	298(2)
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/n$	$C2/c$	$P2_1/n$
a (Å)	4.8050(6)	11.208(1)	18.215(2)	13.056(2)
b (Å)	22.413(3)	18.930(2)	24.601(2)	8.275(1)
c (Å)	10.875(2)	13.124(2)	11.556(1)	13.056(2)
β (°)	102.399(2)	98.426(2)	103.588(2)	104.100(3)
V (Å ³)	1143.8(3)	2754.4(5)	5033.2(9)	1368.1(4)
Z	4	4	8	2
D_{calc} (g cm ⁻³)	1.401	1.491	1.441	1.697
μ (Mo K α) (mm ⁻¹)	0.098	0.682	1.020	0.865
F(000)	504	1280	2240	704
Measured reflections	10430	15995	13037	5483
Unique reflections	2026	5124	4588	2038
Observed reflections ($I \geq 2\sigma(I)$)	1388	4299	3057	1427
Parameters	167	381	340	205
Restraints	1	32	2	14
GOF on F^2	1.032	1.116	0.993	1.015
R_1, wR_2 [$I \geq 2\sigma(I)$] ^a	0.0468, 0.0953	0.0700, 0.2230	0.0496, 0.1254	0.0535, 0.1302
R_1, wR_2 (all data) ^a	0.0819, 0.1124	0.0806, 0.2345	0.0786, 0.1436	0.0853, 0.1435

^a $R_1 = F_o - F_c/F_o$, $wR_2 = [\sum w(F_o^2 - F_c^2)/\sum w(F_o^2)^2]^{1/2}$

Table 2. Selected bond distances (Å) and angles (°) for the complexes

1			
Co(1)–O(1)	1.905(3)	Co(1)–O(3)	1.923(3)
Co(1)–N(1)	1.926(4)	Co(1)–N(2)	1.856(4)
Co(1)–N(4)	1.933(4)	Co(1)–N(5)	1.852(4)
N(5)–Co(1)–N(2)	178.44(18)	N(5)–Co(1)–O(1)	96.03(16)
N(2)–Co(1)–O(1)	82.50(16)	N(5)–Co(1)–O(3)	82.26(16)
N(2)–Co(1)–O(3)	97.18(16)	O(1)–Co(1)–O(3)	89.85(14)
N(5)–Co(1)–N(1)	98.42(17)	N(2)–Co(1)–N(1)	83.04(17)
O(1)–Co(1)–N(1)	165.49(16)	O(3)–Co(1)–N(1)	90.71(16)
N(5)–Co(1)–N(4)	82.78(17)	N(2)–Co(1)–N(4)	97.76(17)
O(1)–Co(1)–N(4)	91.27(16)	O(3)–Co(1)–N(4)	165.04(16)
N(1)–Co(1)–N(4)	91.93(17)		
2			
Zn(1)–O(1)	2.162(3)	Zn(1)–O(3)	2.110(2)
Zn(1)–N(1)	2.230(3)	Zn(1)–N(2)	2.052(3)
Zn(1)–N(4)	2.265(3)	Zn(1)–N(5)	2.047(3)
N(5)–Zn(1)–N(2)	169.45(10)	N(5)–Zn(1)–O(3)	75.49(9)
N(2)–Zn(1)–O(3)	115.05(9)	N(5)–Zn(1)–O(1)	105.23(9)
N(2)–Zn(1)–O(1)	74.78(10)	O(3)–Zn(1)–O(1)	93.20(11)
N(5)–Zn(1)–N(1)	105.21(10)	N(2)–Zn(1)–N(1)	75.15(11)
O(3)–Zn(1)–N(1)	95.16(11)	O(1)–Zn(1)–N(1)	149.55(9)
N(5)–Zn(1)–N(4)	74.64(10)	N(2)–Zn(1)–N(4)	94.81(10)
O(3)–Zn(1)–N(4)	149.95(9)	O(1)–Zn(1)–N(4)	91.46(10)
N(1)–Zn(1)–N(4)	95.71(11)		
3			
Cd(1)–N(1)	2.379(6)	Cd(1)–O(3)	2.396(5)
Cd(1)–N(2)	2.417(5)		
N(1)–Cd(1)–N(1A)	101.9(3)	N(1)–Cd(1)–O(3A)	149.79(19)
N(1)–Cd(1)–O(3)	105.20(18)	O(3)–Cd(1)–O(3A)	53.2(2)
N(1)–Cd(1)–N(2A)	95.1(2)	N(1)–Cd(1)–N(2)	67.7(2)
O(3)–Cd(1)–N(2A)	122.10(19)	O(3)–Cd(1)–N(2)	83.20(18)
N(1)–Cd(1)–N(2A)	95.08(19)	O(3)–Cd(1)–N(2A)	122.10(19)
N(2)–Cd(1)–N(2A)	153.3(3)		

tively, in MeOH. The C, H, N content determined from the elemental analyses agree well with those determined by single-crystal X-ray diffraction techniques.

3. 2. IR Spectra

The weak and broad absorptions centered at 3420–3454 cm^{-1} are attributed to the stretching vibration of the O–H bonds. The sharp bands observed at 3241 cm^{-1} of HL and at 3387 cm^{-1} of complex **3** are attributed to the stretching vibration of the amino N–H bonds. The strong bands at 1628 cm^{-1} of the hydrazone HL and at 1638 cm^{-1} of complex **3** are due to the $\nu(\text{C}=\text{O})$ stretching vibrations, and the bands at 1596–1607 cm^{-1} due to the $\nu(\text{C}=\text{N})$ stretching vibrations. The absence of the $\nu(\text{C}=\text{O})$ bands in the spectra of complexes **1** and **2** suggests the enolization of the amide functionality upon coordination to the metal atoms. For the spectrum of complex **3** typical band for $\nu(\text{C}=\text{O})$ indicates that the carbonyl group is free. The typical bands for the nitrate ligand for complex **3** are observed

at 1456, 1311 and 1072 cm^{-1} , and that for the azide anion is at 2072 cm^{-1} .⁹

3. 3. Structure Description of Complex 1

The structure of complex **1** is presented in Figure 1. The asymmetric unit comprises of a cobalt complex molecule, two methanol molecules and one water molecule. The coordination sphere of the Co atom is a distorted octahedral, with the tridentate hydrazone ligands coordinated in a meridional mode, generating five membered chelate rings with angles in the range 82.26(16)–83.04(17)°. The coordinate bond angles are within normal values.⁹ The hydrazone coordinates to the Co atom with the pyridine nitrogen, enolate oxygen and imino nitrogen atoms. Ligand L is nearly coplanar, with the dihedral angles between the benzene and pyridine rings of 10.7(5) and 13.2(5)°, respectively. The bond lengths are similar to the cobalt complexes containing the enolate form of this ligand type.^{5,10,11}

Figure 1. A perspective view of complex 1 with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level.

3. 4. Structure Description of Complex 2

The structure of complex 2 is presented in Figure 2. The coordination sphere of the Zn atom is a distorted octahedral, with the tridentate hydrazone ligands coordinated in a meridional mode, generating five membered chelate rings with angles in the range 74.78(10)–75.49(9)°. The coordinate bond angles are within normal values.¹² The hydrazone coordinates to the Zn atom with the pyridine nitrogen, imino nitrogen and enolate oxygen atoms. Ligand L is somewhat deviated from planarity, with dihedral angles between the benzene and pyridine rings of 19.1(6) and 11.1(6)°, respectively. The bond lengths are similar to the zinc complexes containing the enolate form of this ligand type.^{12,13}

Figure 2. A perspective view of complex 2 with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level.

3. 5. Structure Description of Complex 3

The structure of complex 3 is presented in Figure 3. The asymmetric unit comprises of a cadmium complex cation and an azide anion. The cadmium complex cation possesses a crystallographic two-fold rotation axis symmetry, with the axis passing through the line of Cd(1)–N(4)–O(4). The coordination sphere of the Cd atom can be described as a severely distorted octahedral, with the bidentate hydrazone ligands coordinated in a meridional mode, generating five a membered chelate ring with angle of 67.7(2)°. The coordinate bond angles are within normal values.¹⁴ The hydrazone coordinates to the Cd atom through the pyridine nitrogen, imino nitrogen and enolate oxygen atoms. Hydrazone ligand is nearly coplanar, with dihedral angles between the benzene and pyridine rings of 7.9(5)°. The nitrate ligand coordinates to the Cd atom through two oxygen atoms. The coordinate bond lengths are comparable to the cadmium complexes containing this ligand type.^{14,15}

Figure 3. A perspective view of complex 3 with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level.

3. 6. Antibacterial Activity

The MIC (Minimum Inhibitory Concentration, $\mu\text{g mL}^{-1}$) values of the compounds were determined by a colorimetric method using the dye MTT.¹⁶ HL and the three complexes were screened for antibacterial activities against two Gram (+) bacterial strains (*B. subtilis* and *S. aureus*) and two Gram (–) bacterial strains (*E. coli* and *P. fluorescence*). The MIC values of the compounds against four bacteria are listed in Table 3. Penicillin G was used as a reference. The hydrazone HL has medium activity against *S. aureus*, weak activity against *B. subtilis*, and no activity against *E. coli* and *P. fluorescence*. The complexes have in general stronger activities against the bacterial strains than the free hydrazone. Complex 1 has strong activity against *B. subtilis* and *S. aureus*, and weak activity against *E. coli* and *P. fluorescence*. Complex 2 has medium

activity against *B. subtilis* and *S. aureus*, weak activity against *P. fluorescence*, and no activity against *E. coli*. Complex **3** has strong activity against all the bacterial strains. The MIC values of complex **3** on *B. subtilis* and *S. aureus* are 2.3 and 1.2 $\mu\text{g mL}^{-1}$, which are better than penicillin G. However, the free hydrazone and the complexes have no activity against two fungal strains *Candida albicans* and *Aspergillus niger*.

Table 3. Antimicrobial activities of the compounds

Tested material	Minimum inhibitory concentrations ($\mu\text{g mL}^{-1}$)			
	<i>B. subtilis</i>	<i>S. aureus</i>	<i>E. coli</i>	<i>P. fluorescence</i>
HL	37.5	18.8	> 150	> 150
1	4.7	9.4	37.5	37.5
2	18.8	18.8	> 150	37.5
3	2.3	1.2	9.4	4.7
Penicillin G	2.3	4.7	>150	>150
DMSO	> 150	> 150	> 150	> 150

4. Conclusion

In summary, three new cobalt(II), zinc(II) and cadmium(II) complexes derived from 2-hydroxy-*N'*-(pyridin-2-ylmethylene)benzohydrazide were prepared and structurally characterized. The metal atoms in the complexes are in octahedral coordination. The cobalt and cadmium complexes have effective antibacterial activities against *Bacillus subtilis*, *Staphylococcus aureus*, *Escherichia coli*, and *Pseudomonas fluorescence*.

5. Supplementary Material

CCDC 1875732 (HL), 1875733 (**1**), 1875734 (**2**), and 1875735 (**3**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at <http://www.ccdc.cam.ac.uk/const/retrieving.html> or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk.

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Povzetek

Tri nove spojine, in sicer kobaltov(II), cinkov(II) in kadmijev(II) kompleks $[\text{CoL}_2] \cdot 2\text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$ (**1**), $[\text{ZnL}_2]$ (**2**) in $[\text{Cd}(\text{HL})_2(\text{NO}_3)_2] \cdot \text{N}_3$ (**3**) smo sintetizirali z uporabo 2-hidroksi-*N'*-(piridin-2-ilmetilen)benzohidrazida (HL) ter jih okarakterizirali z IR, UV-Vis in monokristalno rentgensko difrakcijo. Rentgenska analiza je razkrila, da so kompleksi enojedrne zvrsti s kovinskim ionom v oktaedrični koordinaciji. Hidrazonu in njegovim kompleksom smo določili tudi antibakterijske (*Bacillus subtilis*, *Staphylococcus aureus*, *Escherichia coli*, *Pseudomonas fluorescens*) in antimikotične (*Candida albicans*, *Aspergillus niger*) lastnosti. Kompleksi imajo izražene protibakterijske lastnosti.

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