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Synthesis, Characterization, Crystal Structures, and Biological Activity of Nickel(II), Manganese(II) and Cadmium(II) Complexes Derived from 4-Bromo-*N*'-(1-(Pyridin-2-yl)ethylidene)benzohydrazide

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Abstract—Three nickel(II), manganese(II) and cadmium(II) complexes, [NiL_2] (**I**), [MnL_2] (**II**) and [$Cd(HL)_2(NO_3)$]$\cdot NO_3$ (**III**), derived from 4-bromo-*N*'-(1-(pyridin-2-yl)ethylidene)benzohydrazide (HL) were prepared and characterized by IR and UV spectra. Structures of the complexes were further confirmed by single crystal X-ray diffraction techniques (CIF files CCDC nos. 1874307 (**I**), 1874308 (**II**), and 1874309 (**III**)). The Ni atom in complex **I** and the Mn atom in complex **II** are in octahedral coordination. The Cd atom in complex **III** is in square antiprism coordination. The nickel complex is effective on the inhibition of *Jack bean* urease.

Keywords: hydrazone, complex, crystal structure, biological activity

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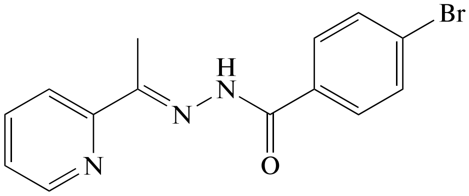
INTRODUCTION

In recent years, much efforts have been focused on hydrazones because they have a wide range of biological activities, such as antibacterial [1, 2], antitumor [3, 4], anti-inflammatory [5, 6], and cytotoxic [7, 8], etc. Some hydrazones have been reported to possess strong urease inhibitory activities [9]. Hydrazones are a kind of versatile ligands during the coordination with metal ions [10–14]. Some vanadium complexes derived from hydrazides show interesting urease inhibitory activities [15]. You and coworkers have reported that some Schiff base complexes are effective urease inhibitors [16–20], and some hydrazones have various biological properties [21–23]. In pursuit of new urease inhibitors, in this work, three nickel(II), manganese(II) and cadmium(II) complexes, [NiL_2] (**I**), [MnL_2] (**II**), and [$Cd(HL)_2(NO_3)$]$\cdot NO_3$ (**III**), derived from 4-bromo-*N*'-(1-(pyridin-2-yl)ethylidene)benzohydrazide (HL, Scheme 1), are reported.



Scheme 1.

EXPERIMENTAL

Materials and methods. 2-Acetylpyridine, 4-bromobenzohydrazide and inorganic metal salts were obtained from TCI. The remaining chemicals were obtained from Xiya Chemical Co. Elemental analyses (CHN) were performed on a Perkin-Elmer 240C elemental analyzer. Infrared spectra were recorded on a Jasco FT/IR-4000 spectrophotometer in the region 4000–400 cm^{-1} using KBr pellets. Electronic absorption spectra were recorded with a Lambda 35 spectrophotometer. ^1H NMR and ^{13}C NMR spectra for HL were recorded on a Bruker spectrometer. Single crystal

X-ray diffraction was carried out with a Bruker SMART 1000 CCD diffractometer.

Caution! Perchlorate salts are potentially explosive. Only a small amount of such material should be prepared, and they should be handled with great care.

Synthesis of HL. 2-Acetylpyridine (1.2 g, 0.010 mol) diluted by methanol (50 mL) was mixed with 4-bromobenzohydrazide (0.21 g, 0.010 mol) dissolved by methanol (50 mL). The mixture was refluxed for 1 h, and the solvent was removed by distillation under reduced pressure. The residue was recrystallized from methanol to yield colorless single crystals. The yield was 2.9 g (76%).

FT-IR data (KBr; ν, cm^{-1}): 3283 ν(NH), 1659 ν(C=O), 1587 ν(C=N). UV data (methanol; λ, nm (ε, mol^{-1} L cm^{-1}): 245 (15.300), 298 (18.100), 365 (7.600). ^{1}H NMR (500 MHz; DMSO-*d*^{6}; δ, ppm): 10.92 (s., 1H, N*H*), 8.61 (d., 1H, Py*H*), 8.11 (d., 1H, Py*H*), 7.84–7.71 (m., 6H, Py*H* + Ar*H*), 2.46 (s., 3H, C*H*$_3$). ^{13}C NMR (126 MHz; DMSO-*d*^{6}; δ, ppm): 163.19, 154.98, 148.56, 147.71, 136.54, 132.04, 130.11, 129.58, 125.22, 124.81, 120.36, 12.63.

For $C_{14}H_{12}N_3OBr$

Anal. calcd., %	C, 52.8	H, 3.8	N, 13.2
Found, %	C, 53.0	H, 3.9	N, 13.0

Synthesis of the complexes. HL (0.32 g, 1.0 mmol) dissolved in methanol (30 mL) was mixed with $Ni(CH_3COO)_2 \cdot 4H_2O$ (0.25 g, 1.0 mmol), $MnCl \cdot 4H_2O$ (0.20 g, 1.0 mmol), and $Cd(NO_3)_2 \cdot 4H_2O$ (0.31 g, 1.0 mmol) for complexes **I**, **II** and **III**, respectively. The mixtures were stirred at room temperature for half an hour and filtered. The filtrate was kept in air for several days to form suitable crystals for single crystal X-ray diffraction.

[NiL$_2$] (**I**). Green block crystals. The yield was 232 mg (33%). FT-IR data (KBr; ν, cm^{-1}): 1650 ν(C=O); 1585 ν(C=N); and 1492, 1450, 1363, 1160, 1072, 945, 855, 777, 752, 682, 546, 516. UV data (methanol; λ, nm (ε, mol^{-1} L cm^{-1}): 285 (11.100), 372 (16.200). Λ_M (10^{-3} M in methanol): 18 Ω^{-1} cm^2 mol^{-1}.

For $C_{28}H_{22}N_6O_2Br_2Ni$

Anal. calcd., %	C, 48.5	H, 3.2	N, 12.1
Found, %	C, 48.6	H, 3.2	N, 12.0

[MnL$_2$] (**II**). Brown block crystals. The yield was 167 mg (24%). FT-IR data (KBr; ν, cm^{-1}): ν(C=O) 1655; ν(C=N) 1583; and 1495, 1448, 1363, 1158, 1070, 945, 851, 750, 678, 629, 544, 512. UV data (methanol; λ, nm (ε, mol^{-1} L cm^{-1}): 298 (13.500), 363 (15.300). Λ_M (10^{-3} M in methanol): 27 Ω^{-1} cm^2 mol^{-1}.

For $C_{28}H_{22}N_6O_2Br_2Mn$

Anal. calcd., %	C, 48.8	H, 3.2	N, 12.2
Found, %	C, 48.9	H, 3.3	N, 12.0

[Cd(HL)$_2$(NO$_3$)]·NO$_3$ (**III**). Colorless block crystals. The yield was 375 mg (43%). FT-IR data (KBr; ν, cm^{-1}): 3122 ν(NH), 1650 ν(C=O); 1592 ν(C=N); 1383 ν(NO$_3$); 1466 ν(ONO$_2$); and 1269, 1129, 1067, 958, 864, 823, 616, 522. UV data (methanol; λ, nm (ε, mol^{-1} L cm^{-1})): 300 (12.300), 367 (13.400). Λ_M (10^{-3} M in methanol): 132 Ω^{-1} cm^2 mol^{-1}.

For $C_{28}H_{24}N_8O_8Br_2Cd$

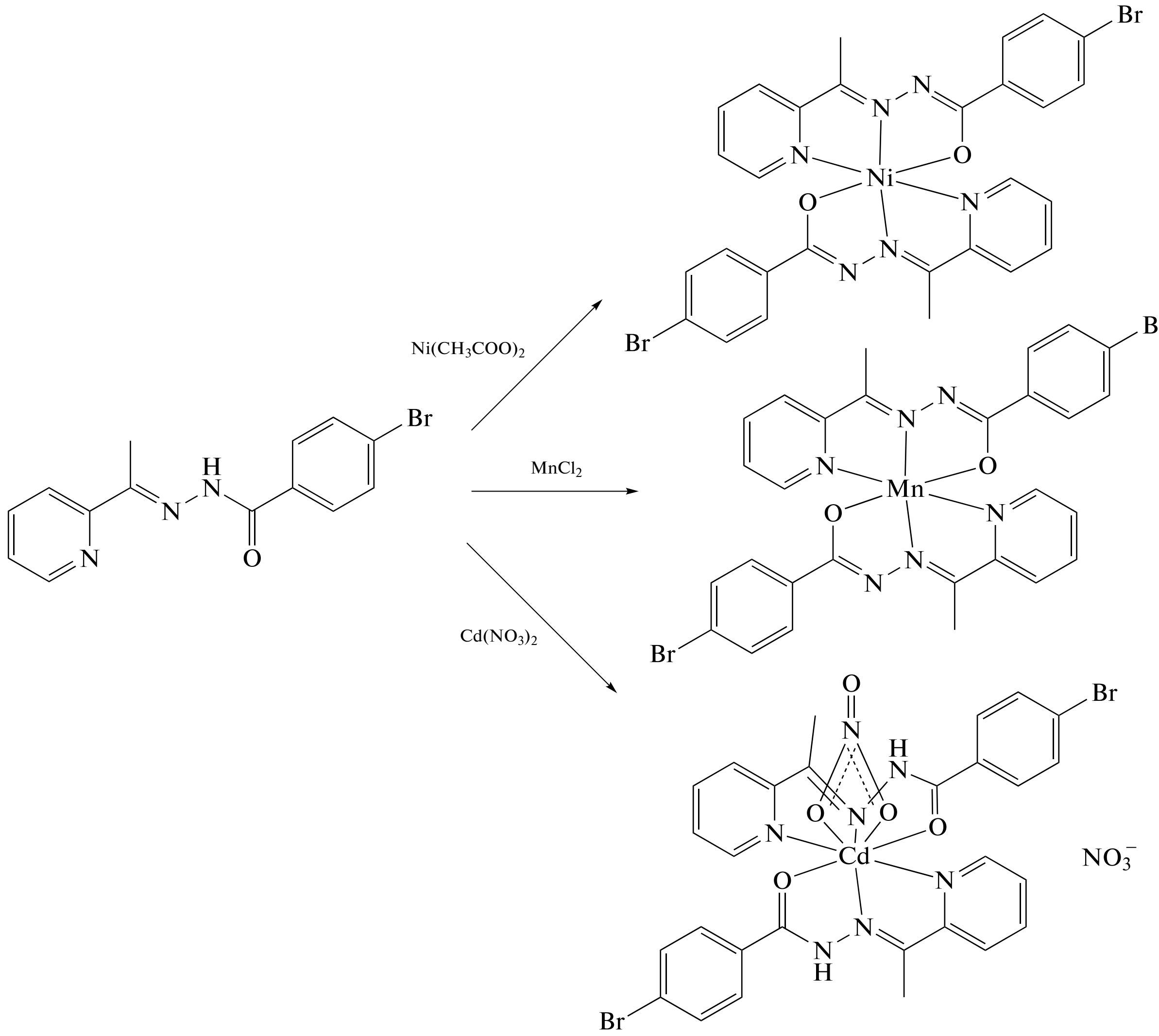
Anal. calcd., %	C, 38.5	H, 2.8	N, 12.8
Found, %	C, 38.7	H, 2.7	N, 13.0

X-ray crystallography. The diffraction data were collected using graphite-monochromatized MoK_α radiation (λ = 0.71073 Å) on a Bruker SMART 1000 CCD diffractometer at 298(2) K. The collected intensity data were reduced with SAINT [24]. The multiscan absorption correction was performed with SADABS [25]. Structures of the complexes were solved with SHELXTL by direct methods and refined against F^2 by full-matrix least-squares method [26]. The nonhydrogen atoms were refined anisotropically. The hydrogen atoms were placed in calculated positions and constrained to ride on their parent atoms. Crystallographic data for the complexes are summarized in Table 1. Selected bond lengths and angles are given in Table 2.

Supplementary material for structures has been deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1874307 (**I**), 1874308 (**II**), and 1874309 (**III**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/conts/retrieving.html).

RESULTS AND DISCUSSION

HL was prepared by 2-acetylpyridine and 4-bromobenzohydrazide in methanol. The complexes were prepared by HL with metal salts in methanol (Scheme 2). Single crystals of the complexes were obtained by slow evaporation of the methanolic solution of the compounds. Crystals of the compounds are stable in common condition. Molar conductivity of the complexes in methanol with 10^{-3} mol L^{-1} are in the range of 18–27 Ω^{-1}cm^2 mol^{-1} for complexes **I** and **II**, and 92 Ω^{-1} cm^2 mol^{-1} for complex **III**, indicates the non-electrolytic nature of complexes **I** and **II**, and 1 : 1 electrolytic nature of complex **III** in solution [27].



Scheme 2.

Molecular structures of complexes **I** and **II** are shown in Fig. 1. Displacement ellipsoids are drawn at the 30% probability level. Atoms labeled with the suffix *A* are related to the symmetry operations $-x$, $1 + y$, $3/2 - z$ for **I** and $-x$, y, $3/2 - z$. for **II**. The molecules of the complexes possess crystallographic two-fold rotation axis symmetry with the axis passes through the metal atoms. The ligands coordinate to the metals with the imino N, pyridine N, and enolate O atoms, generating five-membered chelate rings with bite angles of 76.7(1)° and 78.2(2)° for **I**, and 71.3(2)° and 71.5(2)° for **II**. The metal centers of the two complexes are in octahedral coordination. The three donor atoms of one hydrazone ligand, and the imino N atom of the other hydrazone ligand are located at the equatorial plane, and the pyridine N atom and enolate O atom of the hydrazone ligand are situated at the axial sites. The bond lengths around the metal atom of **I** are shorter than **II**. Even so, they are similar to the reported nickel(II) and manganese(II) complexes with hydrazones [28–31].The octahedral geometries are distorted, which can be found from the *cis* and *trans* bond angles of 76.7(1)°–104.8(1)° for **I**, 71.3(2)°–105.9(2)° for **II**, and 154.8(1)°–176.2(2)° for **I**, 142.2(2)°–175.8(3)° for **II**. The pyridine and benzene rings of the ligands form dihedral angles of 12.9(4)° for I and 11.4(5)° for **II**.

The molecules of the complexes are connected by hydrogen bonds (Table 3) to form 1D chains directed from the *b* axis. There are C–H···π interactions between C(7) atom and the rings Ni(1)–O(1)–C(8)–N(3)–N(2), Ni(1)–O(1*A*)–C(8*A*)–N(3*A*)–N(2*A*), and Ni(1)–N(1*A*)–C(5*A*)–C(6*A*)–N(2*A*) with the distances of 2.94(3), 2.94(3), and 2.90(3) Å, respectively, in **I**. And, there are C–H···π interactions between C(7) atom and the ring N(1)–C(1)–C(2)–C(3)–C(4)–C(5) with the distance of 2.87(3) Å in **II**.

Molecular structure of complex III is shown in Fig. 2. The asymmetric unit of the compound comprises of a mononuclear cadmium complex cation and

Table 1. Crystallographic data for complexes **I–III**

Parameters	Value		
	I	**II**	**III**
Formula	$C_{28}H_{22}N_6O_2Br_2Ni$	$C_{28}H_{22}N_6O_2Br_2Mn$	$C_{28}H_{24}N_8O_8Br_2Cd$
Crystal shape, color	Block, green	Block, brown	Block, colorless
Crystal size, mm	0.27 × 0.23 × 0.23	0.30 × 0.27 × 0.27	0.18 × 0.17 × 0.17
Crystal system	Orthorhombic	Orthorhombic	Monoclinic
Space group	*Pbcn*	*Pbcn*	*P*2/*c*
a, Å	11.9998(18)	12.3046(12)	13.8926(17)
b, Å	9.7873(13)	9.8991(10)	8.6890(11)
c, Å	23.7861(15)	23.305(2)	15.0496(14)
β, deg	90	90	121.122(2)
V, Å^3	2793.6(6)	2838.7(5)	1555.2(3)
ρ_{calcd}, mg/m^3	1.648	1.613	1.864
Z	4	4	2
μ, cm^{-1}	3.592	3.317	3.335
F(000)	1384	1372	860
$\theta_{min}-\theta_{max}$, deg	1.71–25.50	1.75–25.50	1.71–25.48
T_{min}/T_{max}	0.4438/0.4921	0.4361/0.4679	0.5851/0.6010
Observed reflections	15649	16008	8021
Unique reflections (R_{int})	2606 (0.0542)	2634 (0.0579)	4751 (0.0197)
Reflections with $I \geq 2\sigma(I)$	1601	1530	4157
Number of refinementparameters	178	177	427
Restraints	0	0	8
Goodness of fit on F^2	1.010	1.032	1.025
R_1, wR_2 ($I \geq 2\sigma(I)$)	0.0466, 0.1082	0.0716, 0.1684	0.0299, 0.0751
R_1, wR_2 (all data)	0.0896, 0.1319	0.1243, 0.2006	0.0348, 0.0778
Largest diff. peak and hole, e Å^{-3}	0.681 and –0.754	1.162 and –1.123	0.795 and –0.816

a nitrate anion. The ligands coordinate to the Cd atoms through the imino N, pyridine N, and carbonyl O atoms, forming five-membered chelate rings with bite angles of 63.8(1)°–66.4(1)°, which are lower than complex **II**. The Cd atom is coordinated by six donor atoms from two neutral hydrazone ligands, and from two nitrate O atoms. The bond lengths around the metal atoms are similar to the reported cadmium(II) complexes with hydrazones [32, 33]. The pyridine and benzene rings form dihedral angle of 16.2(3)° in the hydrazone ligand. The molecules of the complex are connected by intermolecular hydrogen bonds (Table 3) to form a 3D network.

The N–H absorptions of the free hydrazone and complex **III** are observed at 3283 and 3122 cm^{-1}, respectively. The C=O absorption of the free hydrazone is observed at 1659 cm^{-1}. The C=O absorptions are shifted to lower wavenumbers, viz. 1648–1655 cm^{-1} for the complexes, that indicates they participate in coordination. The C=N absorptions of HL and the complexes are located in the region 1583–1592 cm^{-1} [23]. The bands at 1383 and 1466cm^{-1} of the spectrum of complex **III** can be assigned to the ionic and bridging nitrate [34, 35]. The UV spectra of the free hydrazone and the complexes displayed strong bands in the region 270–300 nm, which are assigned to the π–π*

Table 2. Selected bond lengths (Å) and angles (deg) for complexes **I**–**III**

Bond	*d*, Å	Bond	*d*, Å
I			
Ni(1)–N(2)	1.981(3)	Ni(1)–O(1)	2.081(3)
Ni(1)–N(1)	2.087(4)		
II			
Mn(1)–O(1)	2.146(5)	Mn(1)–N(2)	2.179(5)
Mn(1)–N(1)	2.264(5)		
III			
Cd(1)–N(1)	2.367(3)	Cd(1)–N(2)	2.418(2)
Cd(1)–O(1)	2.561(2)	Cd(1)–O(3)	2.411(3)
Angle	ω, deg	Angle	ω, deg
I			
N(2)Ni(1)N(2*A*)	176.2(2)	N(2)Ni(1)O(1*A*)	100.70(13)
N(2)Ni(1)O(1)	76.74(14)	O(1)Ni(1)O(1*A*)	97.08(17)
N(2)Ni(1)N(1*A*)	104.45(14)	N(2)Ni(1)N(1)	78.24(15)
O(1)Ni(1)N(1*A*)	90.74(13)	O(1)Ni(1)N(1)	154.76(13)
N(1)Ni(1)N(1*A*)	92.3(2)		
II			
O(1)Mn(1)O(1*A*)	102.5(3)	O(1)Mn(1)N(2*A*)	105.8(2)
O(1)Mn(1)N(2)	71.5(2)	N(2)Mn(1)N(2*A*)	175.8(3)
O(1)Mn(1)N(1)	142.3(2)	O(1)Mn(1)N(1*A*)	93.8(2)
N(2)Mn(1)N(1*A*)	111.6(2)	N(2)Mn(1)N(1)	71.4(2)
N(1)Mn(1)N(1*A*)	93.8(2)		
III			
N(1)Cd(1)N(1*A*)	89.3(1)	N(1)Cd(1)O(3)	112.6(1)
N(1)Cd(1)O(3*A*)	152.9(1)	O(3)Cd(1)O(3*A*)	52.8(1)
N(1)Cd(1)N(2*A*)	94.1(1)	O(3)Cd(1)N(2*A*)	124.5(1)
N(1)Cd(1)N(2)	66.4(1)	O(3)Cd(1)N(2)	81.3(1)
N(2)Cd(1)N(2*A*)	153.2(1)	N(1)Cd(1)O(1)	127.2(1)
N(1)Cd(1)O(1*A*)	78.1(1)	O(3)Cd(1)O(1)	75.9(1)
O(3)Cd(1)O(1*A*)	75.0(1)	N(2)Cd(1)O(1*A*)	124.9(1)
N(2)Cd(1)O(1)	63.8(1)	O(1)Cd(1)O(1*A*)	147.4(1)

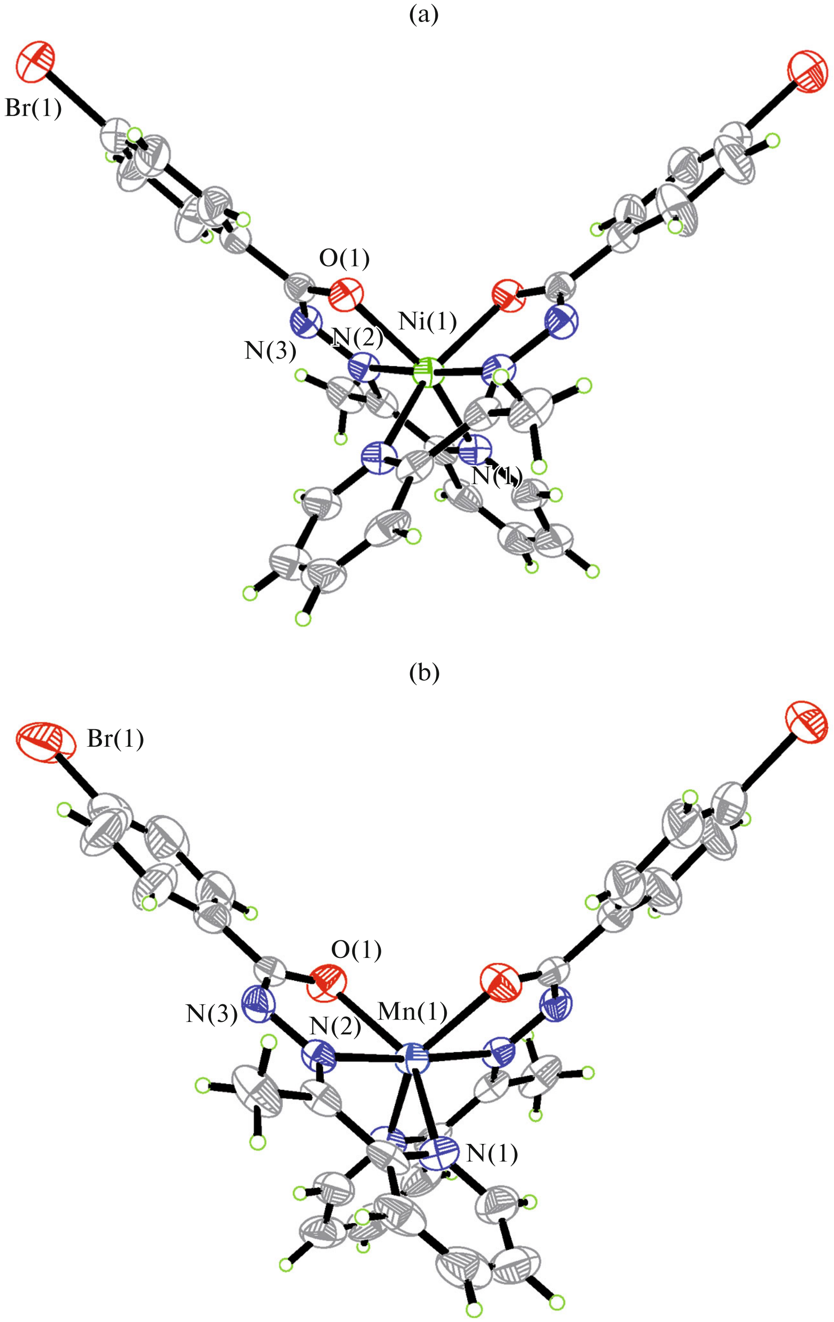


Fig. 1. Molecular structures of **I** (a) and **II** (b), showing the atom-numbering scheme. Hydrogen atoms are omitted for clarity.

transition. The charge transfer LMCT bands of the complexes are located in the region 350–400 nm.

The assay of the urease inhibitory activity was accord to the literature method [36]. The inhibition rate of the compounds (100 μmol L^{-1}) on *Jack bean* urease is given in Table 4. The free hydrazone HL has weak activity. However, the nickel complex **I** has stronger activity than HL with IC_{50} of 8.71 μmol L^{-1}. However, the manganese complex **II** and the cadmium complex **III** have weak activity.

Thus, the present study reports the synthesis, characterization, and crystal structures of a series of new Ni(II), Mn(II), and Cd(II) complexes derived from 4-bromo-*N*'-(1-(pyridin-2-yl)ethylidene)benzohydrazide. X-ray crystallographic study reveals that the Ni and Mn atoms are in octahedral coordination, and the Cd atom is in square antiprism coordi-

Table 3. Geometric parameters of hydrogen for complexes **I–III***

D–H···*A*	Distance, Å			Angle *D*–H···*A*, deg
	D–H	H···*A*	*D*···*A*	
I				
C(4)–H(4)···O(1)$^{\#1}$	0.93	2.30(4)	3.182(5)	157(3)
II				
C(4)–H(4)···O(1)$^{\#2}$	0.93	2.40(3)	3.206(4)	145(5)
III				
N(3)–H(3*B*)···O(6)	0.86	2.24(3)	3.012(4)	150(5)
C(3)–H(3*A*)···O(3)$^{\#3}$	0.93	2.45(3)	3.055(4)	123(5)
C(7)–H(7*A*)···O(6)	0.96	2.57(3)	3.382(4)	142(5)
C(7)–H(7*A*)···O(7)$^{\#4}$	0.96	2.55(3)	3.136(4)	120(5)
C(7)–H(7*B*)···O(1)$^{\#5}$	0.96	2.60(3)	3.470(4)	152(5)
C(14)–H(14)···O(7)	0.93	2.35(3)	3.190(4)	150(5)

*Symmetry codes: $^{\#1}$ $1/2 + x, -1/2 + y, 1/2 - z$; $^{\#2}$ $-1/2 + x, -1/2 + y, 1/2 - z$; $^{\#3}$ $-x, -y, -z$; $^{\#4}$ $1 - x, y, 1/2 - z$; $^{\#5}$ $x, -y, -1/2 + z$.

nation. The nickel complex has effective urease inhibitory activity.

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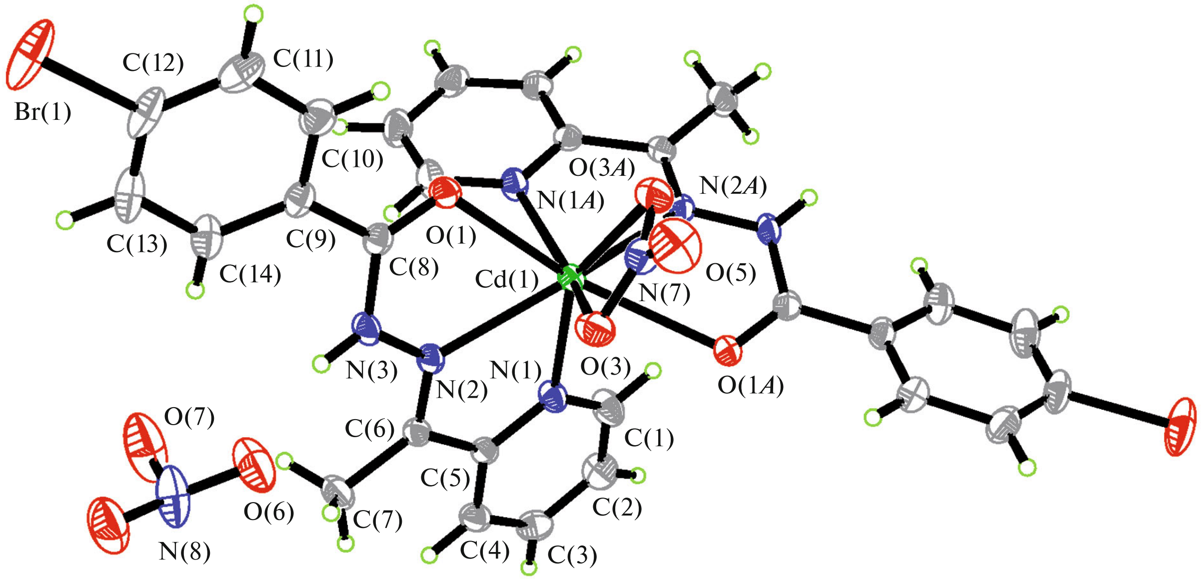

Fig. 2. Molecular structure of **III**, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Table 4. Inhibition of urease by the tested materials

Tested materials	Percentage inhibition*	IC$_{50}$, μmol L^{-1}
HL	13.5 ± 1.9	>100
I	87.2 ± 3.0	8.71 ± 1.53
II	26.3 ± 2.5	>100
III	40.2 ± 2.3	>100
Nickel perchlorate	23.3 ± 2.9	>100
Manganese acetate	16.5 ± 1.8	>100
Cadmium nitrate	93.4 ± 3.4	18.3 ± 2.0
Acetohydroxamic acid	83.7 ± 3.1	29.5 ± 1.7

* The concentration of the tested material is 100 μmol L^{-1}.

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