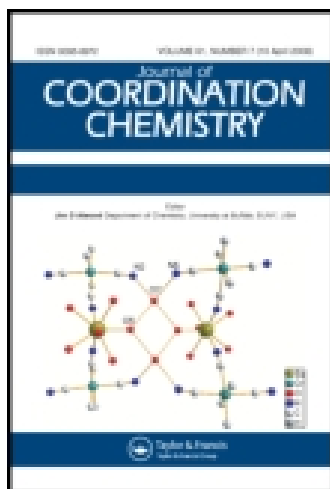


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A 2-D nickel coordination polymer with an unsymmetric ligand, 5-(3-pyridyl)-1,3,4-oxadiazole-2-thione

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A 2-D nickel coordination polymer with an unsymmetric ligand, 5-(3-pyridyl)-1,3,4-oxadiazole-2-thione

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A new nickel coordination polymer was obtained from an unsymmetric building block 5-(3-pyridyl)-1,3,4-oxadiazole-2-thione (**HL6**) and $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ -afforded $[\text{Ni}(\text{L6})_2(\text{H}_2\text{O})_2]_n$ (**1**) with a (4,4) network. Complex **1** exhibits moderate antimicrobial activity against *Bacillus subtilis* ATCC 6633 and *Candida albicans* ATCC 90028. Thermogravimetric data and magnetic moments for **1** have been investigated.

Keywords: Nickel coordination polymer; 5-(3-Pyridyl)-1,3,4-oxadiazole-2-thione; Unsymmetric ligand; 2-D network; Topology

1. Introduction

Metal coordination polymers attract considerable attention because they possess optical, electrical, magnetic, gas absorption and separation, and catalytic properties [1–9], as well as their intriguing architectures and molecular topologies. Bridging ligands play an important role in coordination frameworks with tunable properties and functions. Multi-dimensional metal coordination polymers based on 1,3,4-oxadiazole back-bone display fascinating structures and exhibit interesting magnetic, luminescent, and gas absorption properties [10–12]. A few metal complexes containing 5-pyridyl-1,3,4-oxadiazole-2-thione possess unique structures and intriguing properties [11a, 12a,b, 13]. However, coordination chemistry of 5-(3-pyridyl)-1,3,4-oxadiazole-2-thione (**HL6**) has not been extensively explored compared to bridging ligands containing 5-(4-pyridyl)-1,3,4-oxadiazole-2-thione and 5-(2-pyridyl)-1,3,4-oxadiazole-2-thione [12a,b, 13, 14]. Additionally, 1,3,4-oxadiazole derivatives exhibit some biological activities [15]. Studies on biological activities of metal-containing oxadiazole derivatives remain sparse despite some related compounds possessing such properties [16a]; some metal coordination compounds with biological activities have been obtained [16b–e].

Recently, we reported the crystal structures and properties of metal coordination complexes containing 5-pyridyl-1,3,4-oxadiazole-2-thione ligand after designing and

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self-assembling coordination polymers constructed from unsymmetric ligands [10b, 11a, 12a, 14a, 17]. As a part of our continuing studies on metal coordination complexes containing **HL6**, herein we report the synthesis, crystal structure, and properties of $[\text{Ni}(\text{L6})_2(\text{H}_2\text{O})_2]_n$ (**1**).

2. Experimental

2.1. Materials and physical measurements

All starting materials and solvents were obtained commercially and used as received. Elemental analyses were performed on a Perkin–Elmer 240 elemental analyzer. The FT-IR spectra were recorded from KBr pellets at 400–4000 cm^{-1} on a Bruker Tensor spectrometer. Thermogravimetric analysis (TGA) data were collected with a Perkin–Elmer TGS-2 analyzer in N_2 at a heating rate of 10 $^\circ\text{C min}^{-1}$.

2.2. Synthesis of $[\text{Ni}(\text{L6})_2(\text{H}_2\text{O})_2]_n$ (**1**)

To 5 mL aqueous solution of sodium hydroxide (20 mg, 0.5 mmol), 85 mg (0.5 mmol) of **HL6** was added. The mixture was stirred until **HL6** dissolved. The final solution was filtered and diluted with 2 mL of *N,N*-dimethylformamide (DMF). Then 5 mL of water/DMF solution (1/1; v/v) of nickel nitrate (73 mg, 0.25 mmol) was added slowly. The reaction mixture was stirred for 1 h, then filtered and left to stand at room temperature. Block dark-green crystals of **1** were obtained in 1 month in 65% yield (73 mg). Anal. Calcd for $\text{C}_{14}\text{H}_{12}\text{N}_6\text{NiO}_4\text{S}_2$: C, 37.27%; H, 2.68%; N, 18.63%. Found: C, 37.47%; H, 2.67%; N, 18.68%. IR (KBr pellet, cm^{-1}): 3301(br), 3082(w), 1667(m), 1617(s), 1580(m), 1489(s), 1397(s), 1250(w), 1212(w), 1194(w), 1165(m), 1122(w), 1088(s), 1037(w), 1024(w), 972(w), 937(w), 822(m), 700(m), 672(m), 645(m), 598(m), 544(m), 413(w).

2.3. X-ray crystallography

Single crystal X-ray diffraction of **1** was carried out with a Bruker APEX II CCD diffractometer equipped with a graphite crystal monochromator situated in the incident beam for data collection at 298(2) K. The lattice parameters were obtained by least-squares refinement of the diffraction data of reflections, and data collections were performed with Mo- $\text{K}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). All the measured independent reflections were used in the structural analysis and semi-empirical absorption corrections were applied using SADABS. The program SAINT [18] was used for integration of the diffraction profiles. All structures were solved by direct methods using the SHELXS program of the SHELXTL package and refined with SHELXL [19]. Metal atoms were located from the *E*-maps and other non-hydrogen atoms were located in successive difference Fourier syntheses. The final refinement was performed by full-matrix least-squares with anisotropic thermal parameters for all the non-hydrogen atoms based on F^2 . All hydrogens were first found in difference electron density maps, and then placed in calculated sites and included in the final refinement in

Table 1. Crystal data and structure refinement parameters for **1**.

Empirical formula	C ₁₄ H ₁₂ N ₆ NiO ₄ S ₂
Formula weight	451.13
Temperature (K)	293
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>
Unit cell dimensions (Å, °)	
<i>a</i>	6.9713(2)
<i>b</i>	9.5303(3)
<i>c</i>	13.5647(4)
β	98.031(2)
Volume (Å ³), <i>Z</i>	892.38(5), 2
Calculated density (g cm ⁻³)	1.679
Absorption coefficient (mm ⁻¹)	1.356
Crystal size (mm ³)	0.04 × 0.05 × 0.18
θ range for data collection	2.6–27.5
Reflections collected	6357
Independent reflections	2048 [<i>R</i> (int) = 0.057]
Goodness of fit on <i>F</i> ²	1.005
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	1293 <i>R</i> ₁ ^a = 0.0463, <i>wR</i> ₂ ^b = 0.0925
<i>R</i> indices (all data)	<i>R</i> ₁ ^a = 0.0929, <i>wR</i> ₂ ^b = 0.1084
Largest difference peak and hole (e · Å ⁻³)	0.46 and –0.57

^a*R*₁ = $\sum ||F_o| - |F_c|| / \sum |F_o|$. ^b*wR*₂ = $[\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$.

Table 2. Selected bond lengths (Å) and angles (°) for **1**.

Ni1–O2	2.096(4)	Ni1–N3 ^a	2.210(3)
Ni1–N1	2.168(3)	S1–C1	1.688(4)
O2–Ni1–N1	91.66(13)	N1–Ni1–N3 ^a	89.65(10)
O2–Ni1–N3 ^a	90.60(13)	N1–Ni1–N1 ^b	180.00
O2–Ni1–O2 ^b	180.00	N1–Ni1–N3 ^c	90.35(10)
O2–Ni1–N1 ^b	88.34(13)	N3 ^a –Ni1–N3 ^c	180.00
O2–Ni1–N3 ^c	89.41(13)		

Symmetry codes: **1**: ^a1 – *x*, 1/2 + *y*, 3/2 – *z*; ^b1 – *x*, –*y*, 1 – *z*; ^c*x*, –1/2 – *y*, –1/2 + *z*.

the riding model approximation with displacement parameters derived from the parent atoms to which they were bonded. The crystal data as well as details of data collection and refinement for **1** are given in table 1. Selected bond lengths and angles for **1** are listed in table 2.

3. Results and discussion

3.1. Preparation of **1**

Compound **1** was obtained through reaction of **HL6** and nickel nitrate in water and DMF by slow evaporation in air at room temperature. Compound **1** is stable in air and insoluble in water and in common organic solvents. The complex was identified by single-crystal X-ray diffraction, powder X-ray diffraction (PXRD) (Supplementary material), elemental analysis (EA), infrared spectroscopy (IR), and TGA. The crystalline phase purity of the sample was independently confirmed by PXRD, EA, and IR. To further investigate how reaction conditions affect the product, we carefully

examined the ratio of metal ion toward ligand and different metal salts as starting material. When the ratio was fixed at 1 : 2, complex **1** was obtained in moderate yield, but the yield decreased when the ratios of metal ion : ligand changed from 3 : 1 to 1 : 3. These results imply that excess of metal influences on the yield. When other nickel salts were used, the same polymer was achieved, demonstrating that the counter-anions do not affect the product.

3.2. IR spectrum

The FT-IR spectrum of **1** exhibits a strong and broad absorption centered at 3301 cm^{-1} , ascribed to the presence of coordinated water molecule in the structure. Bands at 1617, 1194, 1165, and 1088 cm^{-1} indicate the presence of oxadiazole in **1**. Characteristic bands of C–S and pyridyl can be observed at 937 and 645 cm^{-1} , respectively.

3.3. Description of crystal structure

Single X-ray analysis reveals that **1** is a neutral 2-D framework constructed by Ni(II) centers and **L6**; **L6** adopts μ_2 bridging (nitrogen atoms from pyridyl and oxadiazole). As shown in figure 1, the local coordination environment around Ni(II) shows that each nickel is in a slightly distorted octahedron formed by two identical oxadiazole groups [$\text{Ni}-\text{N}_{\text{oz}} = 2.168(3)\text{ \AA}$] and two pyridyl nitrogens [$\text{Ni}-\text{N}_{\text{py}} = 2.210(3)\text{ \AA}$]. Axial positions of the octahedron are occupied by water [$\text{Ni}-\text{O} = 2.096(4)\text{ \AA}$]. Each **L6** employs both the oxadiazole nitrogen and pyridyl nitrogen to bridge two Ni(II) ions, and each nickel

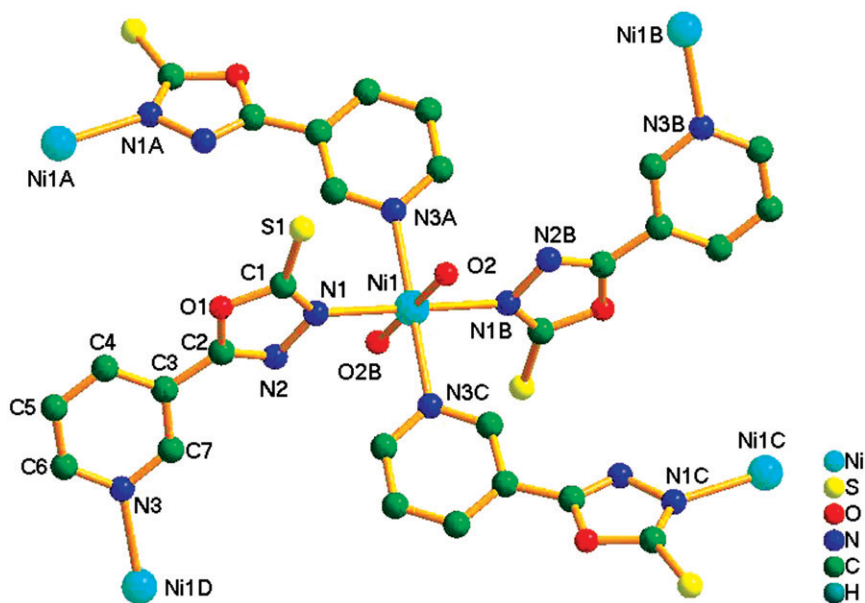


Figure 1. Local coordination environment around the nickel atom for **1** (hydrogen atoms are omitted for clarity).

connects four **L6** ligands, similar to those documented previously [12b]. As a result, a (4,4) 2-D rhombic grid is formed with diagonal measurement of each grid *ca* $9.53 \times 13.56 \text{ \AA}$ based on the metal...metal distances; internal angles of each grid are 109.82° and 70.18° (figure 2a,b). All nickel ions in 2-D frameworks are located in the same plane, similar to that found previously [12a]. Similar to literature, C-S bond of **L6** in **1** is longer than that of free ligand **HL6**, indicating **L6** adopts thiolate form [12b]. The present work is in agreement with previously reported, based on similar system of 1,3,4-oxadiazole-thione/thiolate [10a,c, 12a,b, 14].

There is no interpenetration between adjacent layers and 2-D sheets were packed by the sequence of *AA* along the *ab* plane (figure 2c, table 3), similar to those reported previously [12a, 20]. In **1**, large void spaces do not exist to accommodate guest or solvent molecules, different from those with diverse interpenetrations filling the void space [21]. Comparing to previous work [12a], the present work indicates that the nature of bridging ligands affect the structures and properties of products.

The magnetic moment of **1** was examined at 300 K showing μ_{eff} close to 2.97 B.M. at 300 K, much larger than the expected spin-only value (2.83 B.M.) of one magnetically isolated high-spin Ni(II) ion ($S = 1$).

3.4. Thermogravimetric analysis

TGA of **1**, from 293 to 973 K at 10 K min^{-1} under nitrogen, shows that it loses 8.16% of its weight from 305 to 405 K, ascribed to loss of two coordinated waters (Calcd 7.99%). Then **L6** starts to decompose until 702 K and the remaining product weight is 16.80%, assigned to NiO residue (Calcd 16.56%).

3.5. Antimicrobial activity

Since **1** contains a 1,3,4-oxadiazole group with biological activity, preliminary bioassay was examined. Compound **1** completely inhibited the growth of *Bacillus subtilis* ATCC 6633 and *Candida albicans* ATCC 90028 with minimum inhibitory concentration (MIC) of 100 and $50 \mu\text{g mL}^{-1}$, respectively. Compared to the antimicrobial activity of free **HL6** reported previously, **1** is much more potent [22], which can be attributed to coordination [23]. The antimicrobial activity of **1** was lower than commercial antimicrobial agent, ampicillin (MIC of $25 \mu\text{g mL}^{-1}$). The present result is similar to that found previously [16d, 24].

4. Conclusions

A 2-D nickel(II) coordination polymer **1** with (4,4) topology has been synthesized, characterized, and exhibits moderate antimicrobial activity against *B. subtilis* ATCC 6633 and *C. albicans* ATCC 90028. The present result shows that complex constructed by ligands containing oxadiazole group has potent activity, opening use of metal coordination polymers with biological activities. Further investigation on the preparation and biological property of other metal coordination polymers with such organic ligands is in progress in our laboratory.

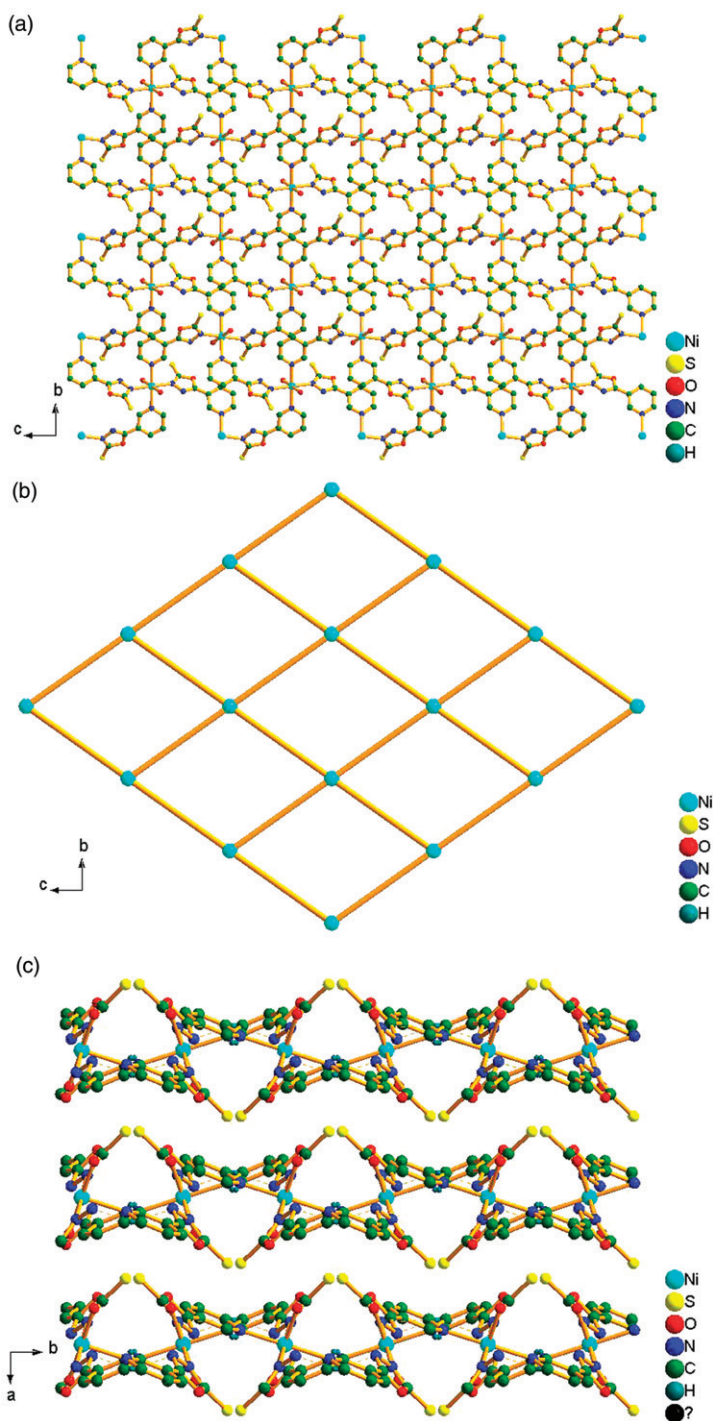


Figure 2. (a) The 2-D layer structure along the bc plane in 1. (b) Schemed topology structure in 1. (c) The perspective of inter-layers along the ab plane in 1.

Table 3. Hydrogen bond geometries in the crystal structure of **1**.

Complex	D–H...A ^a	H...A (Å)	D...A (Å)	D–H...A (°)
	C7–H7...N1 ^a	2.48 (4)	3.100 (5)	127 (3)
	O2–H2WA...N2 ^b	2.35 (6)	2.924 (5)	122 (5)
	C6–H6...N1 ^c	2.59 (5)	3.183 (5)	122 (3)
	C6–H6...N2 ^c	2.28 (5)	3.126 (5)	149 (4)

Symmetry codes: ^a1 – x, – 1/2 + y, 3/2 – z; ^b1 – x, – y, 1 – z; ^cx, – 1/2 – y, 1/2 + z.

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC reference number is 670653 for **1**. Copies of this information may be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>). Supplementary data associated with this article can be found in the online version.

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