Journal of Coordination Chemistry

Crystal structure and magnetic properties of Co(II) coordination polymer with CsCl-like topology

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Accepted author version posted online: 05 Feb 2013.Published
online: 21 Mar 2013.

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Crystal structure and magnetic properties of Co(II) coordination polymer with CsCl-like topology

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One Co(II) coordination polymer with 4-hydroxypyridine-2,6-dicarboxylic acid (H_3L)-$[Co_{1.5}L(H_2O)_4]_n$ (**1**) has been fabricated under hydrothermal condition through reaction of $Co(OH)_2$ and H_3L with mole ratio of 1 : 1. Single-crystal X-ray diffraction analysis reveals that **1** crystallizes in triclinic space group $P\bar{1}$ with a 1-D stair-like structure and rich hydrogen bonding interactions expand the 1-D structure to a CsCl-like 8-connected topology. Magnetic properties of **1** have been studied showing antiferromagnetic exchange interaction between Co(II) ions.

Keywords: Co(II) coordination polymer; Stair-like; CsCl-like topology; Magnetic property

1. Introduction

Engineering of coordination polymers (CPs) has drawn great interest in supramolecular and materials chemistry [1–8], owing to the challenge to design intriguing architectures with potential applications in magnetism, optics, catalysis, and separation [9–16]. Preparation of CPs can be achieved by careful selection of appropriate linkers; in general, polydentate ligands with rigidity and symmetry are suitable to construct CPs [17]. Carboxyl group plays an important role in constructing CPs, owing to flexible coordination, such as terminal monodentate to one metal, bridging bidentate in a syn–syn, syn–anti, and anti–anti configuration to two metals, and bridging tridentate to two metals [18]. Thus, pyridine carboxylic ligands, which combine the pyridine ring (rigid) and carboxyl group (flexible), are employed in construction of CPs, and a large number of CPs containing such ligands have been reported [19–31]. Thus, pyridine-2,6-dicarboxylic acid with a rigid 120° angle between the central pyridine ring and two carboxyl groups has attracted much interest in constructing CPs and has been used to generate polymeric coordination frameworks with homometals as well as heterometals with interesting magnetic and luminescent properties [25–31]. 4-Hydroxypyridine-2,6-dicarboxylic acid (H_3L) contains the merits of pyridine-2,6-dicarboxylic acid. Furthermore, it has a hydroxyl group at the opposite site of N, providing more coordination geometries than pyridine-2,6-dicarboxylic acid [32–35]. Comparing with CPs of pyridine-2,6-carboxylic acid, those of H_3L are relatively limited.

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Among transition metal ions, Co(II) has special interest for magnetic material [36–46], because the paramagnetic Co(II) is favorable to introduce strong magnetic anisotropy to give spin canting, metamagnetism, slow magnetic relaxation, and long-range order [44, 45]. Cobalt(II) has variable coordination numbers from 4 to 6 [46].

Thus, we chose Co(II) to fabricate CPs by reaction of $Co(OH)_2$ with H_3L under hydrothermal condition giving $[Co_{1.5}(L)(H_2O)_4]_n$ (**1**), which is characterized by single X-ray crystallography and magnetic measurements.

2. Experimental

2.1. *Materials and physical measurement*

All reagents and solvents were purchased commercially and used without purification. Elemental analyses for C, H, and N were carried out on a Perkin–Elmer analyzer at the Institute of Elemento-Organic Chemistry, Nankai University. FTIR spectra were recorded from KBr pellets (4000–400 cm^{-1}) on a Bruker tensor 27 FTIR spectrophotometer. Variable-temperature magnetic susceptibilities were measured on a Quantum Design MPMS-7 SQUID magnetometer.

2.2. *Preparation of $[Co_{1.5}L(H_2O)_4]_n$ (1)*

Mixture of 0.3 mmol $Co(OH)_2$ (0.028 g), 0.3 mmol H_3L (0.055 g), and 10 mL H_2O was placed in a 25 mL Teflon-lined stainless steel autoclave, heated at 160 °C for 3 days, and then cooled to room temperature. Red block crystals were obtained in 70% yield based on $Co(OH)_2$. Anal. Calcd for $C_7H_{10}Co_{1.50}NO_9$ (**1**): C, 24.69; H, 2.96; N, 4.11%. Found: C, 24.12; H, 2.77; N, 4.30%. IR (KBr, cm^{-1}): 3420 (s,b), 1602 (s), 1436 (m), 1354 (m), 1082 (m), 1030 (m), 790 (m), 730 (m), 535 (m), and 419 (m).

2.3. *Determination of crystal structure*

Suitable single crystal of **1** was glued on a glass fiber. Data were collected on a BRUKER SMART 1000 CCD detector with graphite-monochromated Mo-K radiation ($\lambda = 0.71073$ Å). Intensity data were collected at 293 ± 2 K. The structure was solved by direct methods using SHELXS-97 [47] and subsequent Fourier difference techniques, and refined anisotropically by full-matrix least-squares on F^2 using SHELXL-97 [48]. The hydrogens were set in calculated positions and refined as riding with common fixed isotropic thermal parameters. Detailed information about crystal data and structure refinement are summarized in table 1. Selected bond lengths and angles of **1** are given in table 2.

3. Results and discussion

Compound **1** was self-assembled through hydrothermal synthesis, exhibiting different structure than other Co(II) carboxylate complexes [36–43], which gives different magnetic properties. Other Co(II) carboxylate complexes are zero, 1-D, 2-D, or 3-D structures; shortest connections between Co(II) ions are –OCO– bridges, compared with O bridges in

Table 1. Data collection and processing parameters for **1**.

Formula	$C_7H_{10}NO_9Co_{1.5}$
M_r [g mol^{-1}]	340.55
T [K]	293(2)
Crystal system	Triclinic
Space group	$P\bar{1}$
a [Å]	7.3072(15)
b [Å]	8.4313(17)
c [Å]	9.3806(19)
α [°]	110.14(3)
β [°]	101.01(3)
γ [°]	95.14(3)
V [Å^3]	524.77(18)
Z	2
ρ_{calcd} [g cm^{-3}]	2.155
μ [mm^{-1}]	2.444
$F(000)$	343
Crystal size [mm]	$0.20 \times 0.20 \times 0.20$
θ [°]	2.38–27.79
Limiting indices	$-9 \leqslant h \leqslant 9$ $-11 \leqslant k \leqslant 10$ $-12 \leqslant l \leqslant 11$
Refl. collected/unique [R(int)]	4924/2407 [0.0312]
Max. and Min. transmission	1.0000 and 0.8971
Data/restraints/parameters	2407/8/170
GOF on F^2	1.016
R_1, wR_2 [$I > 2\sigma(I)$]	0.0271, 0.0608
R_1, wR_2 [all data]	0.0337, 0.0721
Largest diff. peak and hole [e Å^{-3}]	0.402 and -0.637

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

Co1–O9^i	2.075(12)	Co2–N1	2.032(32)
Co1–O9	2.075(12)	Co2–O6	2.035(44)
Co1–O5^i	2.083(55)	Co2–O3^{ii}	2.084(27)
Co1–O5	2.083(55)	Co2–O7	2.101(31)
Co1–O8	2.127(29)	Co2–O2	2.19(4)
Co1–O8^i	2.127(29)	Co2–O3	2.253(29)
O9^i–Co1–O9	180.00(7)	N1–Co2–O6	109.01(7)
O9^i–Co1–O5^i	92.88(6)	N1–Co2–O3^{ii}	145.97(7)
O9–Co1–O5^i	87.12(6)	O6–Co2–O3^{ii}	85.61(7)
O9^i–Co1–O5	87.12(6)	N1–Co2–O7	94.72(7)
O9–Co1–O5	92.88(6)	O6–Co2–O7	151.63(6)
O5^i–Co1–O5	179.98(6)	O3^{ii}–Co2–O7	82.94(6)
O9^i–Co1–O8	92.48(6)	N1–Co2–O2	76.07(7)
O9–Co1–O8	87.52(7)	O6–Co2–O2	87.05(6)
O5^i–Co1–O8	90.40(6)	O3^{ii}–Co2–O2	136.73(6)
O5–Co1–O8	89.60(6)	O7–Co2–O2	83.73(6)
O9^i–Co1–O8^i	87.52(7)	N1–Co2–O3	73.65(6)
O9–Co1–O8^i	92.48(6)	O6–Co2–O3	95.47(6)
O5^i–Co1–O8^i	89.60(6)	O3^{ii}–Co2–O3	74.50(6)
O5–Co1–O8^i	90.40(6)	O7–Co2–O3	106.25(6)
O8–Co1–O8^i	179.99(6)	O2–Co2–O3	148.73(6)

Note: Symmetry codes: (i) $-x$, $-y$, $-z$; (ii) $1-x$, $2-y$, $1-z$.

1, which are much longer. With short bridges between Co(II) ions, there may be interesting magnetic interactions. Furthermore, the hydroxyl in L provides new coordination mode, resulting in a 1-D stair-chain structure.

3.1. *Structural determination of 1*

The single-crystal X-ray diffraction analysis reveals that **1** crystallizes in triclinic space group $P\bar{1}$ with two crystallographically independent Co(II) ions in the asymmetric unit of **1**. Co1 is coordinated with six oxygens, forming an elongated octahedral geometry; O5, O5A, O9, and O9A from two waters and two hydroxyls form the equatorial plane with bond lengths of 2.083(55) (Co1–O5) and 2.075(12) (Co1–O9) and the apical sites are occupied by O8 and O8A of two waters at 2.127(29) (figure 1). The average Co1–O distance is 2.095 Å. Although the coordination number of Co2 is the same as that of Co1, the coordination geometry of Co2 is a seriously distorted octahedron, while NO_2 donors from one L^{3-} and one carboxylate O from another L^{3-} form the equatorial plane and two waters occupying apical sites. The average Co2–O distance (2.133 Å) is slightly larger than that of Co1–O and the Co2–N distance is 2.032(2) Å. Two Co2 ions are bridged by two carboxylate O to form a binuclear $[Co_2O_2]$ grid with angles of 105.50(6)° and 74.50 (6)°, and those $[Co_2O_2]$ grids further connect to each other by Co1 ions through L^{3-}, forming a stair-like chain, as shown in figure 2. The Co2···Co2 and Co2···Co1 distances are 3.454 and 7.437 Å, respectively.

   The carboxylic and carboxyl oxygens of L^{3-} form intermolecular hydrogen bonds with coordinated water, as shown in figure 3; hydrogen bonding parameters are listed in table 3. Each Co1 has eight $[Co_2O_2]$ grids as neighbors (figure 3, left), while each $[Co_2O_2]$ grid is surrounded by eight Co1 ions (figure 3, right). As a result of this connection, a 3-D hydrogen-bonded network is formed (figure 4). Considering $[Co_2O_2]$ and Co1 as 8-connected
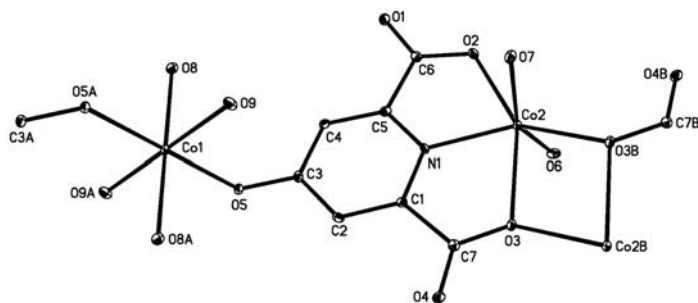

Figure 1.   The coordination environments of two Co(II) ions in **1**. Symmetry codes: (A) −x, −y, −z; (B) 1 − x, 2 − y, 1 − z.
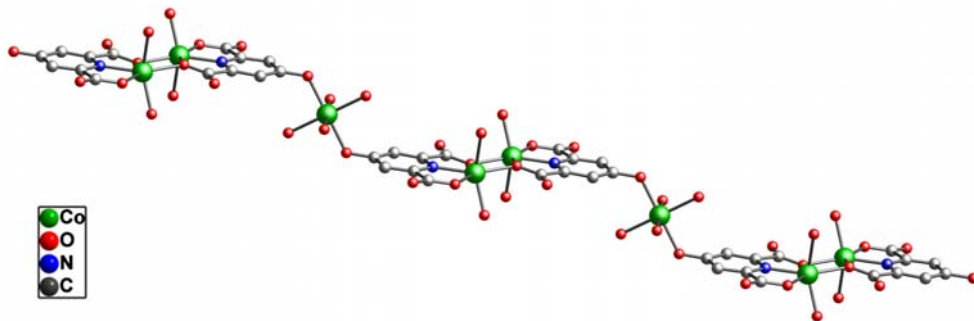

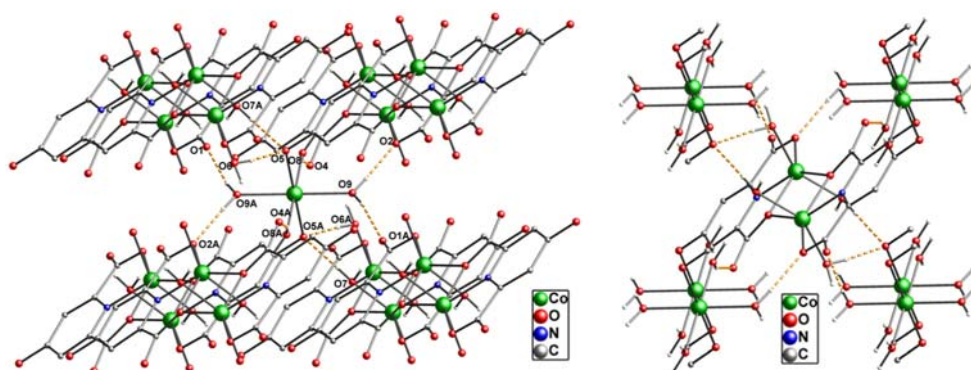Figure 2.   The 1-D stair-like chain of **1**.

Figure 3. The intermolecular hydrogen interactions in **1** (left: eight [Co_2O_2] around one Co1; right: eight Co1 around one [Co_2O_2]). Yellow dotted lines: O–H···O hydrogen bonds. (see http://dx.doi.org/10.1080/00206814.2013.772594 for color version.)

Table 3. Hydrogen bonding parameters in **1**.

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)
O6–H6B···O5^v	0.845(11)	1.790(11)	2.619(15)	174.84(12)
O7–H7B···O5^{viii}	0.849(12)	1.906(27)	2.755(39)	177.86(12)
O8–H8A···O4^{ix}	0.844(20)	1.937(34)	2.757(54)	163.63(11)
O9–H9A···O2^{vii}	0.850(11)	1.887(23)	2.735(34)	175.39(12)
O9–H9B···O1^{iv}	0.854(15)	1.910(28)	2.736(44)	162.40(12)

Note: Symmetry codes: (v) $1 - x$, $1 - y$, $1 - z$; (vi) x, $1 + y$, $1 + z$; (vii) $1 - x$, $1 - y$, $-z$; (viii) x, $1 + y$, z; and (ix) x, $-1 + y$, $-1 + z$.
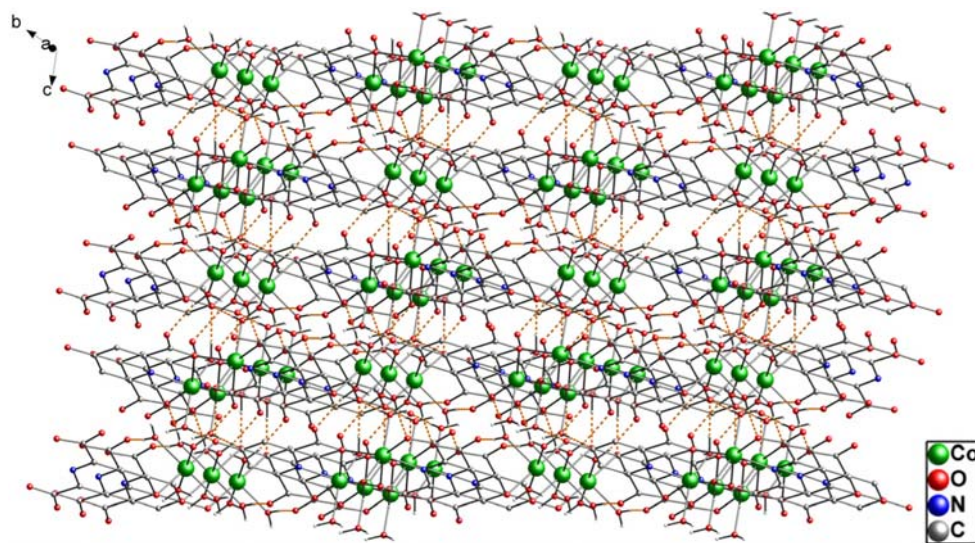
Figure 4. The 3-D network of **1** connected by hydrogen bonds. Yellow dotted lines: O–H···O hydrogen bonds. (see http://dx.doi.org/10.1080/00206814.2013.772594 for color version.)
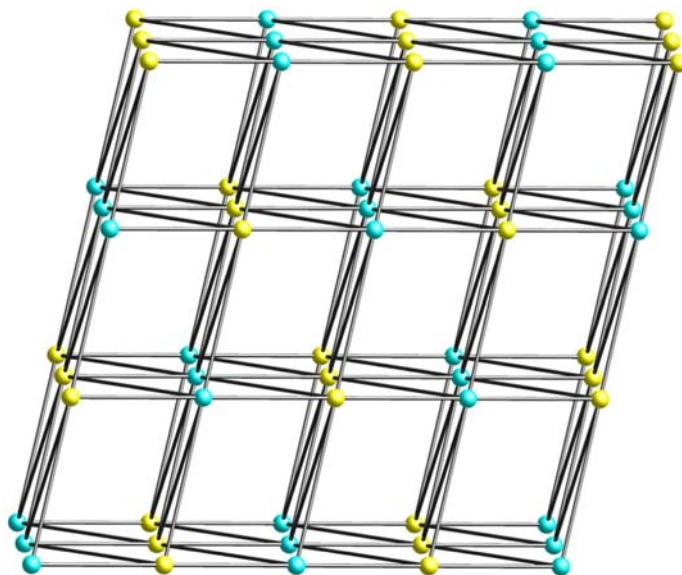
Figure 5.   The CsCl-like 8-connected topology of **1**. Cyan: Co1; yellow: [Co$_2$O$_2$] units. (see http://dx.doi.org/10.1080/00206814.2013.772594 for color version.)

nodes, the 3-D structure can be analyzed as a CsCl-like 8-connected topology [49–51]. Figure 5 with point symbol ($4^{24} \cdot 6^4$), which corresponds to the bcu network.

3.2. *Magnetism*

Compound **1** exhibits a chain structure consisting of one isolated Co1 and a Co$_2$ dimer, which are arrayed alternately. In the Co$_2$ dimer, there are two short O bridges between Co (II) ions, and two building units are bridged by L^{3-}. As a result of this connection, the magnetic interaction should be between Co(II) ions within the dimer. Considering the structure of **1**, the temperature dependence of the magnetic susceptibilities for **1** is measured under applied field of 1000 Oe from 2 to 300 K as shown in the plot of χ_M^T versus *T* (figure 6). The χ_M^T value at 300 K is 1.89 cm^3 K mol^{-1}, which is consistent with the spin-only value of one high-spin Co(II) with $S = 3/2$ and $g = 2$ (1.87 cm^3 K mol^{-1}). With cooling, the χ_M^T decreases slowly to 1.01 cm^3 K mol^{-1} at 2 K due to both the spin-orbit coupling and exchange interactions between adjacent Co(II) ions [52, 53]. The temperature dependence of χ_M^{-1} above 50 K approximates Curie–Weiss behavior with a Curie constant *C* of 2.06 cm^3 K mol^{-1} and Weiss constant θ of –27.32 K. For the spin-orbit coupling contribution of Co(II), we could not definitely explain that interaction occurs between adjacent Co(II) ions. To estimate the strength of the magnetic exchange interactions using the approach for a 1-D Co^{2+} system proposed by Rueff *et al.* [54, 55], the equation below was used to fit the experimental magnetic data.

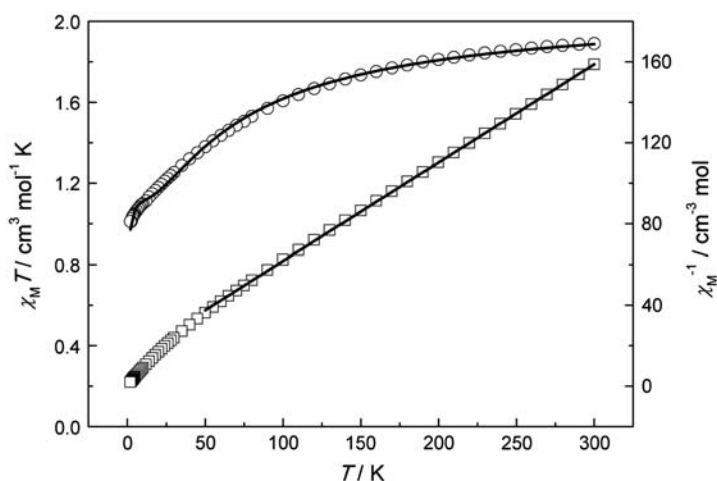$$\chi_M^T = A \exp(-E_1/kT) + B \exp(-E_2/kT)$$

Figure 6. Temperature dependence of χ_M^T (○) and χ_M^{-1} (□) for **1** (solid line represents the best fit indicated in the text).

$A + B$ equals the Curie constant, and E_1 and E_2 represent the "activation energies" corresponding to the spin-orbit coupling and the antiferromagnetic exchange interaction, respectively. The best fitted parameters obtained are, $A + B = 2.07\,\mathrm{cm^3\ K\,mol^{-1}}$, $E_1/\mathrm{k} = 67.04\,\mathrm{K}$, and $E_2/\mathrm{k} = 0.33\,\mathrm{K}$. Therefore, according to the used chain approximation, $\chi_M^T \propto \exp(J/2kT)$, the antiferromagnetic exchange interaction is very weak ($E_2/\mathrm{k} = 0.33\,\mathrm{K}$), corresponding to $J = -0.66\,\mathrm{K}$, which is of the same magnitude as those reported for 1-D Co(II) complexes [56–61]. Taking the structure into account, the negative J value may correspond to the exchange interaction between Co(II) ions within the dimer.

4. Conclusion

A new Co(II) CP (**1**) was produced under hydrothermal condition, adopting chelidamic acid as ligand. It exhibits a stair-like chain and the 3-D hydrogen-bonding network is a CsCl-like 8-connected topology. The experimental fitting indicates that there are weak antiferromagnetic interactions between Co(II) ions.

Supplementary material

The crystallographic data of **1** has been deposited at the Cambridge Crystallographic Data Center as supplementary publications (CCDC-**1**, 648130). These data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html.

Acknowledgments

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