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A new 2-D cobalt coordination polymer with the flexible 2-(1*H*-imidazole-1-yl)acetate: synthesis, structure, and characterization

Yong-Tao Wang^a; Gui-Mei Tang^a; Yong-Chun Zhang^a; Wen-Zhu Wan^a; Jian-Chao Yu^a; Tian-Duo Li^a; Yue-Zhi Cui^a

^a Department of Chemical Engineering, Shandong Institute of Light Industry, Jinan, 250353, P.R. China

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A new 2-D cobalt coordination polymer with the flexible 2-(1*H*-imidazole-1-yl)acetate: synthesis, structure, and characterization

YONG-TAO WANG*, GUI-MEI TANG, YONG-CHUN ZHANG,
WEN-ZHU WAN, JIAN-CHAO YU, TIAN-DUO LI and YUE-ZHI CUI

Department of Chemical Engineering, Shandong Institute of Light Industry,
Jinan, 250353, P.R. China

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A new cobalt coordination polymer with linking unit 2-(1*H*-imidazole-1-yl)acetate (ima), [Co(ima)₂]_n (**1**), has been synthesized and structurally characterized by elemental analysis, IR spectra, and X-ray single-crystal diffraction. The X-ray analysis indicates that a neutral 2-D coordination polymer with (3,6) topology was obtained, which shows a 3-D supramolecular network through C–H···O weak interactions. Complex **1** displays mild antimicrobial activity against *Achromobacter xylosoxidans* ATCC 2706, *Bacillus subtilis* ATCC 6633, and *Candida albicans* ATCC 90028, respectively. The magnetic and thermal gravimetric properties for **1** have been investigated and discussed.

Keywords: Metal coordination polymer; Coordination modes; Cobalt complex; 2-D; (3,6) Topology; 2-(1*H*-imidazole-1-yl)acetate

1. Introduction

Metal coordination polymers have attracted considerable attention in supramolecular chemistry and crystal engineering due to their beautiful structures [1] and potential applications for gas absorption [2], catalysis [3], optoelectronics [4], and magnetic materials [5]. However, the crystal engineering of metal coordination polymers with desired topologies and specific properties still remains a challenge because many factors influence the outcomes, such as coordination geometry, the ligand, the metal-to-ligand ratio, the solvent, etc. [6]. In general, the design of ligands and the coordination preferences of metal ions are most important in manipulating coordination polymer topologies and thus modifying the properties of these materials. A variety of metal–organic networks have been constructed by metal ions and organic ligands containing two or more carboxylate [7], *N*-heterocyclic [8] groups, among which the most intriguing are the porous materials exhibiting potential gas absorption and/or catalytic properties. However, the coordination chemistry of the ligands containing both imidazole and carboxylate remains rarely explored [9]. Imidazole derivatives exhibit

*Corresponding author. Email: ceswyt@sohu.com

biological activities [10] although metal-containing imidazole and carboxylate groups remain less developed; some complexes with biological activities have been investigated and reported [11].

As a continuation of our work on the design of metal coordination polymers based on asymmetric ligands [12], some metal coordination polymers containing 2-(1*H*-imidazole-1-yl)acetate (scheme 1) have been synthesized and characterized, which possess some specific features of tunable topology [9a, c]. Here, we report the synthesis and crystal structure of $[\text{Co}(\text{ima})]_n$ (**1**). In **1**, Co(II) ions are connected by imidazolate and carboxylate to generate 2-D (3,6) sheets, and adjacent sheets are linked further by weak intermolecular hydrogen-bonds to give the 3-D supramolecular framework. The antimicrobial activity, magnetism, IR spectra, and thermogravimetric analysis (TGA) are presented. To the best of our knowledge, this is the first time to investigate antimicrobial activity of a metal complex containing ima.

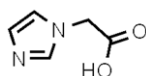
2. Experimental

2.1. Materials and general methods

All the starting materials and solvents for syntheses were obtained commercially and used as received. Elemental analyses were performed on a Perkin-Elmer 240 elemental analyzer. FT-IR spectra were recorded from KBr pellets at 400–4000 cm^{-1} on a Bruker Tensor 27 spectrometer. TGA data were collected with a Perkin-Elmer TGS-2 analyzer in N_2 at a heating rate of 10 $^\circ\text{C min}^{-1}$. Polycrystals of **1** were used to collect the variable temperature magnetic susceptibility $\chi(T)$ data from 2 to 300 K in a magnetic field of 0.1 T using a Quantum Design SQUID magnetometer.

2.2. Synthesis of $[\text{Co}(\text{ima})_2]_n$ (**1**)

Hima (50 mg, 0.4 mmol) and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (58 mg, 0.2 mmol) were mixed in water (10 mL). A dilute aqueous solution of NaOH was added with stirring to adjust the pH of the mixture to 7. The mixture was stirred for 30 min and then filtered. Upon slow evaporation of filtrate at room temperature, well-shaped purple crystals of **1** suitable for X-ray diffraction were obtained within 1 week in 70% yield. Anal. Calcd (%) for $\text{C}_{10}\text{H}_{10}\text{CoN}_4\text{O}_4$: C, 38.85; H, 3.26; N, 18.12. Found (%): C, 38.63; H, 3.28; N, 18.01. IR (cm^{-1}): 3138 m, 1633 vs, 1580 s, 1502 vs, 1526 m, 1384 vs, 1312 m, 1238 m, 1107 m, 1093 m, 1038 m, 974 m, 946 m, 834 m, 792 m, 747 m, 698 vs, 659 m, 623 m, 587 m.



Scheme 1. Ligand of Hima.

2.3. X-ray crystallography

Single crystal X-ray diffraction measurements of **1** were 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 reflection data, and data collections were performed with Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$). All measured independent reflections were used in the structural analysis, and semi-empirical absorption corrections were applied using SADABS; SAINT [13] was used for the integration of the diffraction profiles. The structure was determined by direct methods using the SHELXS program of SHELXTL and refined with SHELXL [14]. Cobalt was 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 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 the riding model approximation with displacement parameters derived from the parent atoms to which they were bonded. The crystal data as well as the details of data collection and refinement for **1** are given in table 1.

3. Results and discussion

3.1. Preparation of **1**

Complex **1** was obtained from aqueous solutions by the reaction of Hima with $\text{Co}(\text{NO}_3)_2$ salts. To further detail the reaction conditions, the ratio of metal ion and ligand and different metal salts as starting material were examined. When the ratio was fixed at 1 : 2, **1** was obtained in moderate yield; various ratios of metal ion toward ligand from 3 : 1 to 1 : 3 were adopted, but the yield of **1** decreased. When other cobalt salts were used, the same polymer was obtained, indicating that the final products are independent of the counter-anion in the metal salt. The quality of crystals was not improved by different syntheses (table 1).

3.2. IR spectra

IR spectra of **1** show the characteristic strong ν_{as} of carboxylate at 1633 and 1580 cm^{-1} . Strong and broad bands at 1384 cm^{-1} may be attributed to the envelope of $\nu_{\text{s}}(\text{COO})$ and the imidazole ring absorption. Another strong absorption of the imidazole group appears at 1502 cm^{-1} [15].

3.3. Description of crystal structure

Compound **1** in the monoclinic centrosymmetric space group, $P2_1/n$, exhibits a neutral 2-D network. As shown in figure 1, cobalt(II) is octahedral, coordinated by

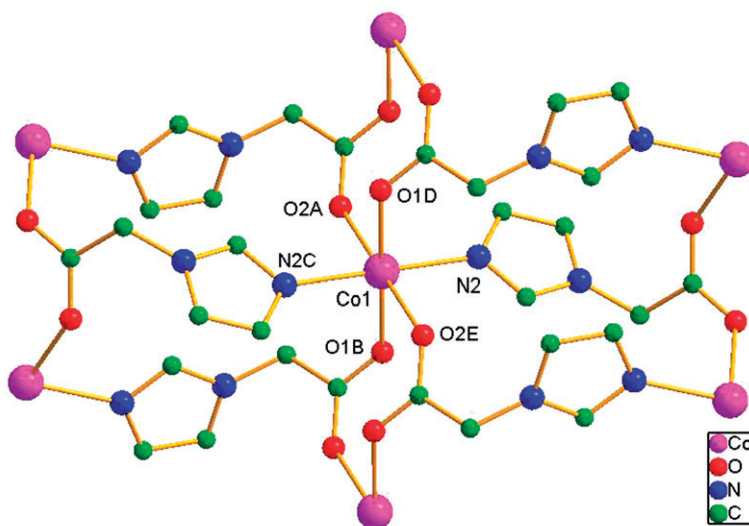
two imidazole nitrogens (N2 and N2C) and four carboxylate oxygens (O1B, O1D, O2A, O2E) from six ima ligands, with the Co–O/N distances being in the range 2.100(10)–2.202(10) Å (table 2). The ima is bonded to three Co's through imidazolate and carboxylates (scheme 2). The imidazoles link Co ions into Co(CON) chains along

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

Empirical formula	C ₁₀ H ₁₀ CoN ₄ O ₄
Formula weight	309.15
Temperature (K)	298
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>n</i>
Unit cell dimensions (Å, °)	
<i>a</i>	8.273(4)
<i>b</i>	5.012(3)
<i>c</i>	12.285(6)
α	90
β	90.908(10)
γ	90
Volume (Å ³), <i>Z</i>	509.3(5), 4
Absorption coefficient (mm ⁻¹)	1.704
Crystal size (mm ³)	0.10 × 0.20 × 0.30
ρ (g cm ⁻³)	2.016
Reflections collected	2304
Independent reflections	885
Reflections [<i>I</i> > 2 σ (<i>I</i>)]	791
<i>F</i> (000)	314
<i>R</i> _{int}	0.090
θ range for data collection (°)	3.0–25.0
Goodness-of-fit on <i>F</i> ²	1.16
Largest hole and peak (e Å ⁻³)	–1.28/2.42
<i>R</i> ₁ ^a / <i>wR</i> ₂ ^b	0.1302/0.3703

$$^a R_1 = \frac{\sum \|F_o\| - |F_c|}{\sum |F_o|}$$

$$^b wR_2 = \frac{[\sum [w(F_o^2 - F_c^2)^2]]^{1/2}}{[\sum w(F_o^2)]^{1/2}}$$

Figure 1. View of the coordination environment of cobalt in **1**.

the *c* direction, with a Co–Co distance of 7.869(3) Å, while carboxylates serve as μ_2 -1,3 bridges in the *syn-anti* mode linking the Co ions into a linear Co(CO₂) chain along the *b* direction, with a Co–Co distance of 5.012(3) Å. These two types of chains intersect each other at Co centers to generate a 2-D flat layer extending along the *bc* plane, and the sheet can be regarded as a (3,6) net with two different linkers (ima and carboxylate) between the metal centers (figure 2a, b) [16]. Such coordination layers are parallel stacked with an *ABAB* sequence (figure 3). Neighboring sheets are further linked by weak intermolecular hydrogen-bond interactions to generate the 3-D metal–organic frameworks (figure 4 and table 3) [17]. The structure of **1** is quite similar to the previously reported Mn(II), Ni(II), and Cd(II) complexes with the same ligand, [M(ima)]_n [9a, c].

3.4. Thermogravimetric analysis

TGA of **1** reveals that the framework is stable up to 300°C and weight loss of 74.2% (Calcd 75.8%) from 308°C to 398°C is attributed to the removal of the ima ligand. The residual percentage weight (Obs. 25.8%) at the end of the decomposition of the complex **1** is in agreement with the formation of CoO (Calcd 24.2%).

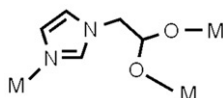
3.5. Antimicrobial activity

Complex **1** is generated by organic ligand containing both imidazole and carboxylic functional groups with biological activity, prompting us to examine its bioassay. Preliminary examination demonstrated that **1** completely inhibits the growth of *Achromobacter xylosoxidans* ATCC 2706, *Bacillus subtilis* ATCC 6633 and *Candida albicans* ATCC 90028 with minimum inhibitory concentration (MIC) of 100, 100, and 50 µg mL⁻¹, respectively (table 4). Compared with the antimicrobial activity of the free ligand reported previously, that of **1** is more potent, which can be attributed to the

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

Co1–N2	2.126(12)	Co1–O1 ^b	2.100(10)
Co1–O2 ^a	2.202(10)		
O2 ^a –Co1–N2	85.2(4)	O1 ^b –Co1–O2 ^a	94.3(4)
O1 ^b –Co1–N2	94.1(4)	O1 ^d –Co1–O2 ^a	85.7(4)
N2–Co1–N2 ^c	180.00	O2 ^a –Co1–O2 ^c	180.00
O1 ^d –Co1–N2	85.9(4)	O1 ^b –Co1–O1 ^d	180.00
O2 ^c –Co1–N2	94.8(4)	O1 ^d –Co1–N2 ^c	94.1(4)
O2 ^c –Co1–N2 ^c	85.2(4)		

Symmetry codes: ^a1/2 – *x*, –1/2 + *y*, 3/2 – *z*; ^b1/2 – *x*, 1/2 + *y*, 3/2 – *z*; ^c1 – *x*, –*y*, 1 – *z*; ^d1/2 + *x*, –1/2 – *y*, –1/2 + *z*; ^e1/2 + *x*, 1/2 – *y*, –1/2 + *z*.



Scheme 2. Coordination modes of ima in **1**.

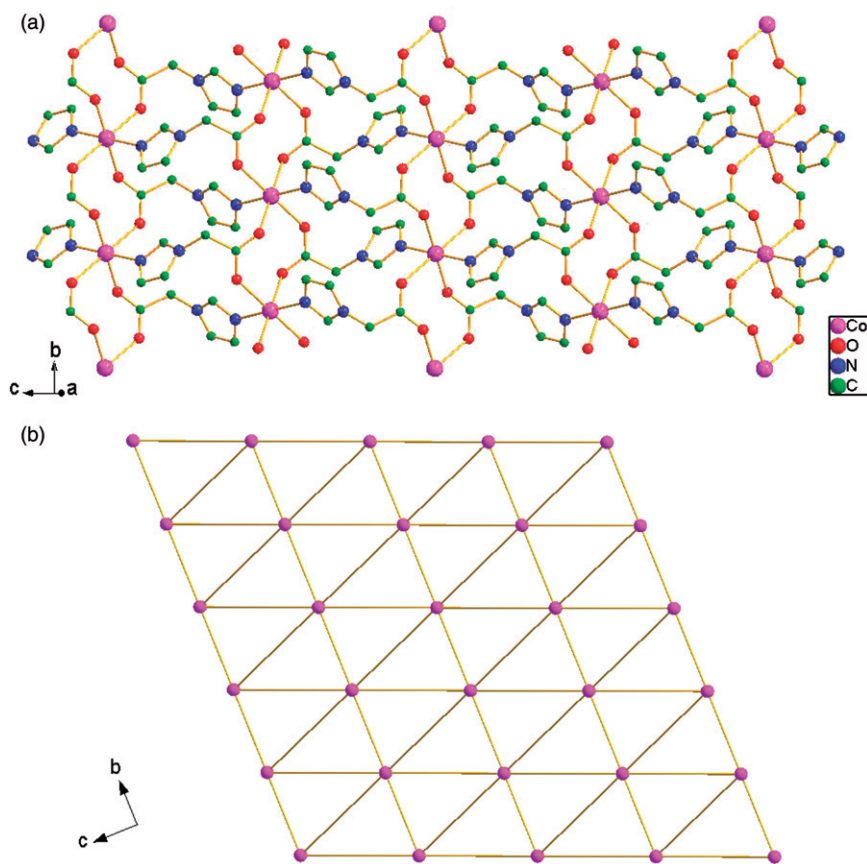


Figure 2. (a) View of 2-D layer along the *bc* plane. (b) The network topology of the 2-D sheet with both carboxylate and imidazolate groups as linkers between nodes (Co's).

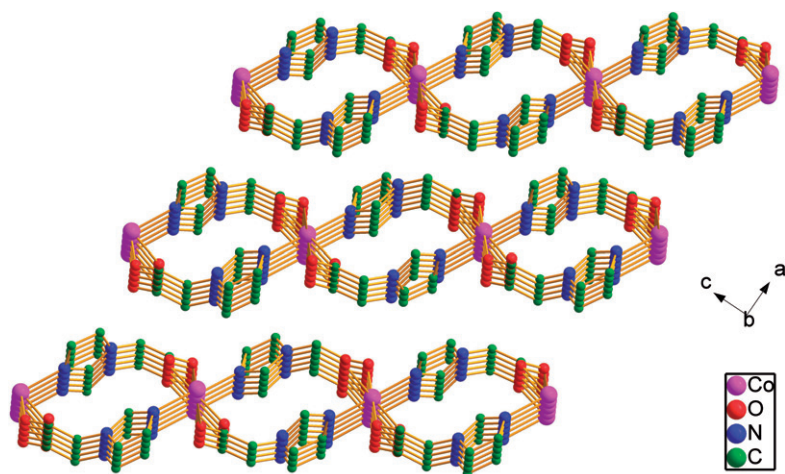


Figure 3. The stacking diagram of the parallel coordination sheets with an *ABAB* sequence along the *b* direction (hydrogen atoms omitted for clarity).

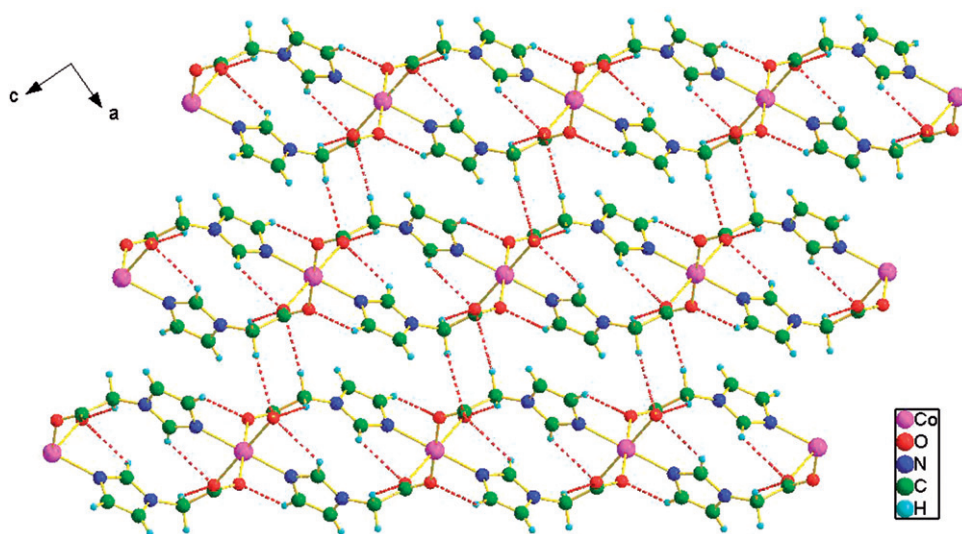


Figure 4. The packing perspective along the [010] plane (the short dash lines stand for the hydrogen bonds).

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

Complex	D–H...A	H...A (Å)	D...A (Å)	D–H...A (°)
1	C3–H3A...O2 ^a	2.500	2.99(2)	114
	C4–H4A...O1 ^b	2.360	3.15(2)	142
	C2–H2A...O2 ^c	2.580	3.41(2)	145
	C2–H2B...O2 ^d	2.590	3.55(2)	170

Symmetry codes: ^a $1/2 - x, -1/2 + y, 3/2 - z$; ^b $1/2 + x, 1/2 - y, -1/2 + z$; ^c $x, -1 + y, z$; ^d $-1/2 - x, -1/2 + y, 3/2 - z$.

Table 4. Antimicrobial activity of **1** (MIC, $\mu\text{g mL}^{-1}$).

Compound	<i>Achromobacter xylosoxidans</i>	<i>Bacillus subtilis</i>	<i>Candida albicans</i>
1	100	100	50
Hima	> 150	> 150	150
Ampicillin	25	25	25

coordination interactions [18]. The antimicrobial activity of **1** was lower than commercial antimicrobial agent, Ampicillin (MIC of $25 \mu\text{g mL}^{-1}$).

3.6. Magnetic property

Magnetic data were collected on crushed single crystals. The temperature dependence of the magnetic susceptibility for **1** is shown in figure 5. The value of $\chi_m T$ at 300 K ($3.38 \text{ cm}^3 \text{ K mol}^{-1}$) is higher than the spin-only value of $1.87 \text{ cm}^3 \text{ K mol}^{-1}$ with $g = 2.0$, expected for uncoupled Co(II) [19, 20]. As T decreases, $\chi_m T$ lowers smoothly and

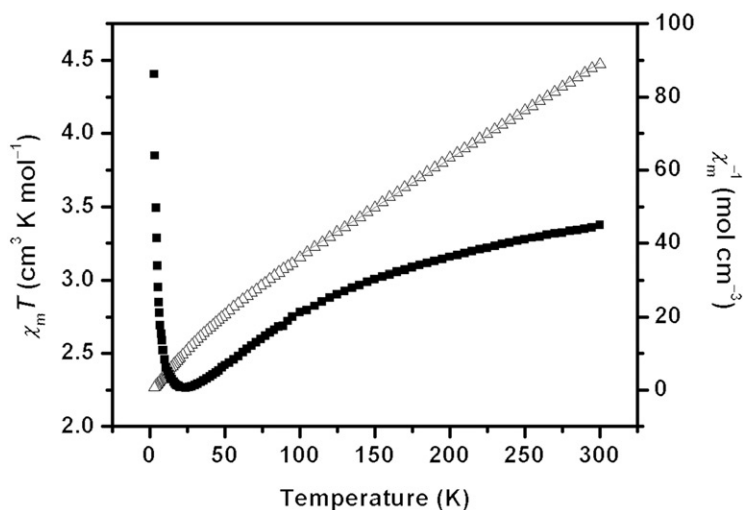


Figure 5. $\chi_m T - T$ and $\chi_m^{-1} - T$ for **1** from 300 to 2 K at 0.1 T.

reaches a rounded minimum around 23.9 K with $\chi_m T$ ($2.26 \text{ cm}^3 \text{ K mol}^{-1}$) and then increases as the T decreases further, a signature of ferrimagnetic response. The $\chi_m T$ product decreases with decreasing temperature in the range 300–100 K, which is a typical behavior of the antiferromagnetically coupled magnetic pair. The magnetic susceptibility in the range 300–100 K follows the Curie–Weiss law with a Curie constant $C = 3.8 \text{ cm}^3 \text{ K mol}^{-1}$ and a Weiss constant θ of -38.4 K , which is a further indication of the antiferromagnetic interactions.

4. Conclusions

A new cobalt coordination polymer with ima containing both imidazolate and carboxylate has been isolated and characterized. Carboxylate as a μ_2 -1,3 bridge in the *syn-anti* mode links Co(II) into interesting 2-D (3,6) topology. Complex **1** exhibits ferromagnetism and has mild antimicrobial activity. We will investigate biological activities of other metal–ima compounds by introducing imidazole and carboxylate.

Supplementary materials

The crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 651850 for **1**. The copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336-033; E-mail: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

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