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Crystal structure of the 2D coordination polymer poly[aqua(μ_2 -2,2'-(1,2-phenylene)diacetato- $\kappa^3 O, O':O'$)-(μ_2 -4,4'-bis((1*H*-1,2,4-triazol-1-yl)methyl)-1,1'-biphenyl- $\kappa^2 N:N'$)cobalt(II)], $C_{28}H_{26}CoN_6O_5$

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Abstract

$C_{28}H_{26}CoN_6O_5$, monoclinic, $P2_1/c$ (no. 14), $a = 13.2393(8)$ Å, $b = 8.6608(5)$ Å, $c = 22.4553(12)$ Å, $\beta = 91.073(2)^\circ$, $V = 2574.3(3)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0327$, $wR_{ref}(F^2) = 0.0804$, $T = 293(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Block, pink
Size:	$0.35 \times 0.31 \times 0.26$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.72 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	25.4° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	16323, 4699, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3577
$N(\text{param})_{\text{refined}}$:	361
Programs:	Bruker programs [1], SHELX [2, 3]

Source of material

All reagents used in these syntheses were of analytical grade and used as purchased without further purification. The mixtures of 1,2-phenylenediacetic acid (phda) (0.1 mmol, 19.4 mg), 4,4'-bis((1*H*-1,2,4-triazol-1-yl)methyl)-1,1'-biphenyl (btmb) (0.1 mmol, 31.6 mg), $\text{Co}(\text{H}_3\text{C}_2\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$ (0.1 mmol, 24.9 mg), NaOH (0.1 mmol), and H_2O (6.0 mL) were placed in a 23 mL Teflon liner stainless steel reactor. The vessel was heated to 393 K for 4 days, and then slowly cooled to room temperature. Pink crystals were obtained, and further crystals were filtered off, washed with mother liquid, and dried under ambient conditions. Yield 46%.

Experimental details

All hydrogen atoms were placed in calculated positions and refined isotropically with a riding model except for those

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Co1	0.42169(2)	−0.00080(3)	0.31041(2)	0.03048(10)
O1	0.43535(11)	−0.05030(16)	0.22128(6)	0.0364(3)
O2	0.34097(12)	0.14089(17)	0.18354(6)	0.0446(4)
O3	0.57726(11)	0.25837(16)	0.16606(6)	0.0427(4)
O4	0.58036(11)	0.43043(18)	0.09544(6)	0.0408(4)
O5W	0.40444(11)	0.23913(15)	0.29330(6)	0.0407(4)
H5WA	0.3791	0.2352	0.2573	0.061*
H5WB	0.4608	0.3090	0.2909	0.061*
N1	0.57931(13)	0.02332(19)	0.32356(7)	0.0366(4)
N2	0.74043(15)	0.0834(3)	0.30574(8)	0.0513(5)
N3	0.73292(13)	0.00854(19)	0.35856(8)	0.0376(4)
N4	1.10765(13)	0.5696(2)	0.77934(8)	0.0380(4)
N5	1.09534(15)	0.4409(3)	0.81235(9)	0.0529(5)
N6	1.25900(13)	0.50492(19)	0.80771(7)	0.0362(4)
C1	0.39275(17)	0.0238(2)	0.17847(9)	0.0343(5)
C2	0.4083(2)	−0.0483(3)	0.11747(9)	0.0538(7)
H2A	0.4778	−0.0828	0.1154	0.065*
H2B	0.3655	−0.1389	0.1141	0.065*
C3	0.38625(17)	0.0550(2)	0.06473(9)	0.0369(5)
C4	0.29045(18)	0.0508(3)	0.03806(10)	0.0459(6)
H4	0.2416	−0.0147	0.0532	0.055*
C5	0.26695(19)	0.1424(3)	−0.01062(10)	0.0517(7)
H5	0.2027	0.1384	−0.0280	0.062*
C6	0.3385(2)	0.2391(3)	−0.03330(10)	0.0526(7)
H6	0.3229	0.3006	−0.0662	0.063*
C7	0.43258(19)	0.2451(3)	−0.00762(9)	0.0444(6)
H7	0.4805	0.3118	−0.0231	0.053*
C8	0.45836(16)	0.1538(2)	0.04108(8)	0.0354(5)
C9	0.56563(17)	0.1650(3)	0.06586(11)	0.0524(7)
H9A	0.5846	0.0672	0.0839	0.063*
H9B	0.6114	0.1851	0.0336	0.063*
C10	0.57573(15)	0.2921(3)	0.11189(9)	0.0352(5)
C11	0.63822(17)	−0.0273(2)	0.36778(10)	0.0415(6)
H11	0.6155	−0.0810	0.4008	0.050*
C12	0.64607(17)	0.0883(3)	0.28686(10)	0.0443(6)
H12	0.6269	0.1333	0.2508	0.053*
C13	0.82243(18)	−0.0323(3)	0.39433(11)	0.0493(6)
H13A	0.8798	−0.0388	0.3682	0.059*
H13B	0.8122	−0.1339	0.4113	0.059*
C14	0.84791(16)	0.0790(3)	0.44417(10)	0.0411(5)
C15	0.78959(18)	0.0859(3)	0.49457(11)	0.0494(6)
H15	0.7309	0.0270	0.4963	0.059*
C16	0.81664(17)	0.1779(3)	0.54209(10)	0.0451(6)
H16	0.7768	0.1782	0.5758	0.054*
C17	0.90186(16)	0.2702(2)	0.54089(9)	0.0373(5)
C18	0.95840(18)	0.2670(3)	0.48959(10)	0.0529(7)
H18	1.0152	0.3297	0.4870	0.063*
C19	0.93207(18)	0.1725(3)	0.44206(10)	0.0538(6)
H19	0.9716	0.1721	0.4083	0.065*
C20	0.93182(16)	0.3656(2)	0.59330(9)	0.0366(5)
C21	0.86153(16)	0.4265(3)	0.63187(10)	0.0427(5)
H21	0.7932	0.4086	0.6245	0.051*
C22	0.89053(17)	0.5130(3)	0.68099(10)	0.0430(6)
H22	0.8417	0.5520	0.7062	0.052*
C23	0.99184(17)	0.5421(2)	0.69304(10)	0.0395(5)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C24	1.06258(17)	0.4819(3)	0.65524(11)	0.0468(6)
H24	1.1309	0.4996	0.6630	0.056*
C25	1.03363(17)	0.3959(3)	0.60604(10)	0.0451(6)
H25	1.0827	0.3573	0.5810	0.054*
C26	1.02305(18)	0.6391(3)	0.74612(10)	0.0481(6)
H26A	1.0423	0.7412	0.7326	0.058*
H26B	0.9660	0.6506	0.7723	0.058*
C27	1.20434(17)	0.6050(3)	0.77703(10)	0.0415(5)
H27	1.2303	0.6890	0.7565	0.050*
C28	1.18794(18)	0.4085(3)	0.82822(10)	0.0486(6)
H28	1.2040	0.3241	0.8522	0.058*

bound to water molecules, which were initially located in a difference Fourier map and included in the final refinement by use of geometrical restraints with the O—H distances being fixed at 0.85 Å and *U*_{iso}(H) equivalent to 1.5 times of *U*_{eq}(O). All calculations were performed using the SHELXL-2014/7 program package [2, 3].

Discussion

As is known, the self-assembly of discrete coordination polymers (CPs) is highly determined by the ligand with certain features such as flexibility, appropriate angles, and versatile binding modes to coordinate to the metal ion with plastic coordination geometry [4–6]. Recently, a large number of CPs have been reported by using mixed-ligand strategy [7–9], most of which was based on the combination of metal-polycarboxylate and *N*-donor molecules, because this strategy can offer chance for new coordination polymers. Among them, benzene multi-carboxylates with long-spanning carboxyl groups has been used by us and others [10, 11], making structures including helices and interpenetrating networks that may be attributed to the flexibility of carboxyl groups. At same time, the dipyriddy-type molecules are widely used coligands that can pillar polycarboxylate motifs into high dimensional structures [12–14].

The asymmetric unit of complex contains one cobalt atom, one *phda* anion, one *btmb* molecule and one ligated water molecule. Each Co atom is octahedrally coordinated by three carboxylate O donors of two symmetry-related *phda* anions and one coordinated water molecule in the equatorial plane with two *N* donors of two symmetry-related *btmb* ligands in the axial positions. The Co—O bond lengths are in the range of 2.0586(15)–2.1963(15) Å and the Co—N bond lengths are 2.1113(19) and 2.1508(19) Å, respectively. Both carboxylate groups of the *phda* ligand bridge adjacent Co(II) centers by a monodentate bridging and a bidentate chelating mode to form zigzag carboxylate chains running along the crystallographic *b* axis. The adjacent

chains are further interconnected to each other through the *btmb* molecules resulting in stepwise (4,4) grids layer. Each layer is corrugated with two *btmb* ligands, two *phda* anion, and four Co atoms forming repeating rhombic grid with a side length of 5.5338(4)–17.7382(8) Å. Furthermore, the presence of coordinated water molecules leads to the formation of extensive H-bonding scheme within the layers (O(5W)–H(2W)···O(1)) and (O(5W)–H(1W)···O(2)). Interestingly, no H-bonds are observed between the layers, which stack in a slightly off-set parallel fashion and are cohered only by the van der Waals forces to accomplish its entire structure.

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