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Crystal structure of diaqua-bis(3,5-dichloroisonicotinato- $\kappa^1 O$)-bis(1,3-di(pyridin-4-yl)propane- $\kappa^1 N$)cobalt(II), $C_{38}H_{36}Cl_4N_6O_6Co$

Table 1: Data collection and handling.

Crystal:	Pink block
Size:	0.36 × 0.31 × 0.27 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.75 mm ⁻¹
Diffractometer, scan mode:	SuperNova, EosS2, ω
$\theta_{\max}$, completeness:	25.5°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10406, 3627, 0.028
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3200
$N(\text{param})_{\text{refined}}$:	250
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3, 4]

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Abstract

$C_{38}H_{36}Cl_4N_6O_6Co$, triclinic, $P\bar{1}$ (no. 2), $a = 9.2687(4)$ Å, $b = 9.7747(6)$ Å, $c = 12.0131(6)$ Å, $\alpha = 87.947(4)^\circ$, $\beta = 82.062(4)^\circ$, $\gamma = 67.258(5)^\circ$, $V = 993.96(10)$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.0397$, $wR_{\text{ref}}(F^2) = 0.0881$, $T = 292.6(3)$ K.

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The 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.

Source of material

All reagents and solvents were commercially available and used as received. The compound was synthesized by the hydrothermal methods. Typically, the starting mixture containing $Co(OAc)_2 \cdot 4H_2O$ (0.1 mmol, 24.9 mg), 3,5-dichloroisonicotinic acid (dciaH) (0.1 mmol, 19.2 mg), 1,3-di(4-pyridyl)propane (bpp) (0.1 mmol, 19.8 mg) and 6 mL of

water was placed in a 23 mL Teflon liner stainless steel reactor. The vessel was heated to 393 K for 4 days, then cooled slowly to the room temperature. Pink blocked crystals were collected by filtration after washing with deionized water, ethanol and acetone, and allowed to dry in air. Yield 52%.

Experimental details

Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program using Intrinsic Phasing and refined with the ShelXL [3] refinement package. All hydrogen atoms were placed in calculated positions with a riding model except for those bound to water molecules, which were initially located in a difference Fourier map and included in the final refinement and $U_{\text{iso}}(H)$ equivalent to 1.5 times of $U_{\text{eq}}(O)$.

Comment

Design and construction of new kinds of coordination polymers (CPs) continue to receive researchers' attention due to their intriguing architectures and topologies [5, 6], and their potential applications in molecular absorption [7], electric conduction [8], optical [9], and many more. Recently, a number of CPs have been obtained by the elaborate selection of a variety of organic N/O/S-donor ligands, such as carboxylate or N-donor ligands [10, 11]. Among them, organic ligands with carboxylate group have attracted attention owing to their characteristics including strong coordination ability, diverse coordination modes, different organic skeletons of carboxylate linkers, and abundant hydrogen-bonding interactions.

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Co1	0.0000	0.5000	1.0000	0.02412(13)
Cl1	−0.05295(9)	0.99612(8)	0.81011(7)	0.0596(2)
Cl2	0.46486(11)	0.49415(10)	0.70631(8)	0.0794(3)
O1	0.2299(2)	0.7095(2)	0.94570(14)	0.0486(5)
O1W	−0.13683(18)	0.45474(17)	0.89209(12)	0.0339(4)
H1WA	−0.0810	0.4186	0.8299	0.051*
H1WB	−0.1960	0.4064	0.9063	0.051*
O2	0.08612(19)	0.61018(17)	0.86711(12)	0.0336(4)
N1	0.1956(2)	0.29802(19)	0.94660(14)	0.0282(4)
N2	0.9846(3)	−0.3375(2)	0.32120(17)	0.0458(6)
N3	0.2639(5)	0.8590(4)	0.5405(2)	0.0830(10)
C1	0.1749(3)	0.1714(2)	0.93624(18)	0.0307(5)
H1	0.0739	0.1720	0.9554	0.037*
C2	0.2954(3)	0.0402(2)	0.89875(18)	0.0335(5)
H2	0.2750	−0.0453	0.8942	0.040*
C3	0.4466(3)	0.0350(2)	0.86786(17)	0.0318(5)
C4	0.4683(3)	0.1661(3)	0.87975(19)	0.0357(6)
H4	0.5680	0.1685	0.8609	0.043*
C5	0.3422(3)	0.2926(3)	0.91940(18)	0.0328(5)
H5	0.3601	0.3788	0.9277	0.039*
C6	0.5803(3)	−0.1060(3)	0.82543(18)	0.0389(6)
H6A	0.5477	−0.1883	0.8431	0.047*
H6B	0.6690	−0.1203	0.8654	0.047*
C7	0.6354(3)	−0.1104(3)	0.69923(18)	0.0371(6)
H7A	0.5477	−0.0949	0.6579	0.045*
H7B	0.6736	−0.0318	0.6806	0.045*
C8	0.7662(3)	−0.2593(3)	0.66638(19)	0.0418(6)
H8A	0.8485	−0.2759	0.7132	0.050*
H8B	0.7242	−0.3356	0.6835	0.050*
C9	0.8406(3)	−0.2793(2)	0.54513(18)	0.0344(6)
C10	0.7534(3)	−0.2593(3)	0.4571(2)	0.0478(7)
H10	0.6438	−0.2242	0.4709	0.057*
C11	0.8300(4)	−0.2918(3)	0.3487(2)	0.0547(8)
H11	0.7690	−0.2807	0.2912	0.066*
C12	1.0671(3)	−0.3515(3)	0.4058(2)	0.0442(6)
H12	1.1760	−0.3808	0.3892	0.053*
C13	1.0005(3)	−0.3248(3)	0.5169(2)	0.0393(6)
H13	1.0646	−0.3379	0.5728	0.047*
C14	0.1718(3)	0.6829(2)	0.86710(18)	0.0311(5)
C15	0.2054(3)	0.7462(3)	0.75318(19)	0.0365(6)
C16	0.1095(3)	0.8852(3)	0.7199(2)	0.0455(7)
C17	0.1432(5)	0.9367(4)	0.6140(3)	0.0689(10)
H17	0.0770	1.0306	0.5942	0.083*
C18	0.3575(5)	0.7270(4)	0.5714(3)	0.0738(11)
H18	0.4440	0.6706	0.5207	0.089*
C19	0.3333(4)	0.6686(3)	0.6753(2)	0.0509(7)

Usually, inclusion of neutral pyridyl-type ligands in the CP can not only adjust the coordination mode of the carboxylate but also induce supramolecular interactions [12]. As a result, by combining the advantages of two types of groups, we selected the 3,5-dichloroisonicotinate (dcia). In fact, the *dcia* ligand is a suitable candidate for assembling CPs.

In contrast to our expectations the title compound is a mononuclear complex. The asymmetric unit of complex contains half a cobalt atom, one *dcia* anion, one *bpp* molecule and one ligated water molecule. Each cobalt atom is six-coordinated by four carboxylate *O* donors of two symmetry-related *dcia* ligands and two coordinated water molecules in the equatorial plane with two *N* donors of two symmetry-related *bpp* ligands in the axial positions. The Co—O bond lengths are 2.1009(14) and 2.1296(15) Å, respectively. The Co—N bond length is 2.1441(18) Å. The [CoO₄N₂] environment is best described as distorted octahedral geometry. Notably, the presence of coordinated water molecule lead to the formation of H-bonding interactions. Besides intramolecular O(1W)—H(1WB)···O(1) hydrogen bonds, intermolecular O(1W)—H(1WA)···N(2) interactions further extend the adjacent cobalt(II) complex to result in a one-dimensional double-stranded chain running along the [111] direction. The adjacent 1D chains are stacked by van der Waals force as into the 3D supramolecular network.

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