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Crystal structure of *catena*-poly[*diaqua*-bis(3,5-dichloropyridine-4-carboxylato- $\kappa^1 O$)-bis(μ_2 -4,4'-bipyridine- $\kappa^2 N:N'$)cobalt(II)], $C_{22}H_{16}Cl_4CoN_4O_6$

Table 1: Data collection and handling.

Crystal:	Pink block
Size:	$0.36 \times 0.29 \times 0.27$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.11 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.5° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7778, 2393, 0.034
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2167
$N(\text{param})_{\text{refined}}$:	176
Programs:	CrysAlisPRO [1], SHELX [2], Olex2 [3]

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Abstract

$C_{22}H_{16}Cl_4CoN_4O_6$, orthorhombic, $P2_12_12$ (no. 18), $a = 10.1837(5)$ Å, $b = 11.2878(6)$ Å, $c = 11.3984(4)$ Å, $V = 1310.26(11)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0400$, $wR_{\text{ref}}(F^2) = 0.0910$, $T = 290(2)$ K.

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A part 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.

Source of material

All chemicals were used as obtained without further purification. A mixture of Hdpa (3,5-dichloropyridine-4-carboxylic acid, 0.2 mmol, 38.1 mg), 4,4'-bpy (4,4'-bipyridine, 0.1 mmol,

15.7 mg), $Co(OAc)_2 \cdot 4 H_2O$ (0.2 mmol, 49.9 mg), H_2O (6.0 mL) and NaOH (0.5 mol/L, 4 drop) was placed in a 23 mL Teflon-lined autoclave, at 393 K for 4 days, and then slowly cooled down. Pink block-shaped crystals were obtained.

Experimental details

Hydrogen atoms bound to carbon atoms were located from the difference map and generated geometrically except for those bound to water molecules which were located from the difference Fourier syntheses.

Comment

Transition complexes are of great interest due to fascinating structural diversities and their application in gas storage, ion exchange, catalysis, magnetism, and luminescence, and so on [4–10]. The organic compound 3,5-dichloropyridine-4-carboxylic acid (Hdpa) can probably provide the potential to enrich the unpredictable and interesting structural and functional diversities of network structures. To our knowledge, there have been a few examples of Hdpa complexes reported to date [10].

The title structure exhibits one dimensional chain structure constructed by $Co(dpa)(H_2O)$ units. The asymmetric unit of compound 1 consists of one half of a Co(II) ion, one dpa molecule, one 4,4'-bpy and one coordinated water, as shown in the Figure. The Co(II) center adopts a distorted octahedral coordination, in which two nitrogen atoms from two different 4,4'-bpy molecules and two oxygens from two different

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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	0.0000	0.5000	0.26173(6)	0.0204(2)
Cl1	0.40749(18)	0.32835(18)	0.46241(13)	0.0641(6)
Cl2	0.36883(15)	0.35459(16)	−0.01205(13)	0.0524(5)
O1	0.1982(3)	0.4469(3)	0.2588(3)	0.0276(7)
O1W	−0.0677(3)	0.3237(3)	0.2668(3)	0.0319(8)
H1WA	−0.1364	0.3149	0.2218	0.08(2)*
H1WB	−0.0156	0.2736	0.2568	0.049(18)*
O2	0.1805(4)	0.2538(4)	0.2139(5)	0.0643(15)
N1	0.0000	0.5000	0.4490(4)	0.0277(13)
N2	0.0000	0.5000	1.0723(4)	0.0257(13)
N3	0.6642(5)	0.2973(5)	0.2033(5)	0.0477(14)
C1	0.1104(5)	0.5125(6)	0.5101(4)	0.0367(13)
H1	0.1888	0.5212	0.4692	0.035(14)*
C2	0.1147(5)	0.5134(7)	0.6319(4)	0.0372(14)
H2	0.1944	0.5228	0.6707	0.046(16)*
C3	0.0000	0.5000	0.6953(5)	0.0226(14)
C4	0.0000	0.5000	0.8265(5)	0.0232(14)
C5	0.1066(5)	0.5415(5)	0.8900(4)	0.0279(13)
H5	0.1800	0.5711	0.8514	0.043(16)*
C6	0.1029(5)	0.5385(4)	1.0109(4)	0.0260(12)
H6	0.1763	0.5649	1.0519	0.025(13)*
C7	0.2420(5)	0.3466(5)	0.2340(5)	0.0334(12)
C8	0.3911(5)	0.3362(5)	0.2244(5)	0.0310(12)
C9	0.4710(5)	0.3232(5)	0.3218(5)	0.0382(14)
C10	0.6075(6)	0.3049(6)	0.3069(6)	0.0457(16)
H10	0.6597	0.2980	0.3735	0.055*
C11	0.5887(6)	0.3122(6)	0.1086(6)	0.0436(16)
H11	0.6272	0.3077	0.0348	0.052*
C12	0.4560(5)	0.3339(5)	0.1163(5)	0.0349(14)

dpa molecules occupy the equatorial plane and two oxygen atoms from two water molecules occupy the axial position. The Co–N bond lengths are 2.135(5) and 2.159(4) Å while the Co–O bond lengths are 2.106(3) Å. The Co atoms are linked by the 4,4'-bpy molecules to generate a one-dimensional chain propagating along the *c* axis direction. The N1 atom and N2 atom of 4,4'-bpy molecules connect two adjacent Co atoms. The N3 atom of dpa anion remains uncoordinated. There exists one hydrogen bond between coordinated water molecules and carboxylate oxygen atoms of dpa (d(O1W...O2): 2.841(6) Å and O–H–O: 149.9°) within the 1D chain, while intermolecular hydrogen bonds between

coordinated water molecules from one chain and the N atoms from the other chain (d(O1W...N3): 2.715(5) Å and O–H–N: 140.0°) connect the adjacent chains to form the 2D layer structure. Individual layers are stacked in a parallel fashion by van der Waals interactions to form the entire 3D network.

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