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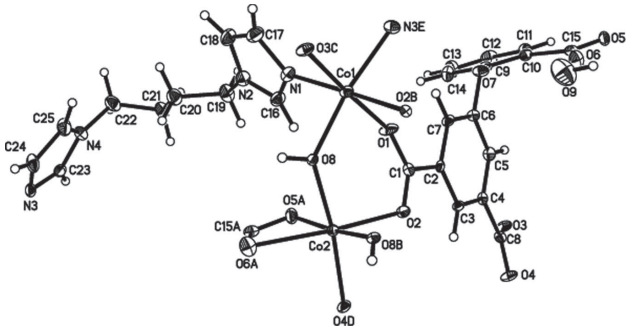
Crystal structure of poly[μ_3-hydroxy-(μ_5-(5-(2-carboxylatophenoxy) isophthalato-$\kappa^6O^1{:}O^2{:}O^3{:}O^4{:}O^5,O^6$)-($\mu_2$-1,4-di(1*H*-imidazol-1-yl)butane-$\kappa^2N{:}N'$)dicobalt(II)] hemihydrate, $C_{25}H_{22}Co_2N_4O_{8.5}$



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Abstract

$C_{25}H_{22}Co_2N_4O_{8.5}$, orthorhombic, *Fdd*2 (no. 43), $a = 19.046(3)$ Å, $b = 45.458(6)$ Å, $c = 11.4233(16)$ Å, $V = 9890(2)$ Å^3, $Z = 16$, $R_{gt}(F) = 0.0339$, $wR_{ref}(F^2) = 0.0618$, $T = 296(2)$ K.

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A part of the polymeric 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

$Co(NO_3)_2 \cdot 6H_2O$ (0.2 mmol), 5-(2-carboxyphenoxy) isophthalic acid (0.1 mmol), 1,4-di(1*H*-imidazol-1-yl)butane (0.1 mmol), DMF (2 mL) and H_2O (4 mL) were placed in a 15 mL Teflon-lined stainless-steel reactor. This mixture was heated at 373 K for 72 h, and then slowly cooled to room temperature. Purple block crystals suitable for X-ray diffraction analysis were collected by filtration directly from the reaction mixture.

Experimental details

The C-bound H atoms were geometrically placed (C—H = 0.93 Å (aromatic), C—H = 0.97 Å (methylene)) and

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Table 1: Data collection and handling.

Crystal:	Block, purple
Size:	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.40 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12388, 3983, 0.052
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3465
$N(param)_{refined}$:	365
Programs:	Bruker programs [1], SHELX [2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.57457(3)	0.54186(2)	0.91020(6)	0.01789(18)
Co2	0.42322(3)	0.51430(2)	1.06775(6)	0.01671(17)
O1	0.48191(17)	0.53881(8)	0.8198(3)	0.0319(10)
O2	0.39981(17)	0.50826(8)	0.8878(3)	0.0204(8)
O3	0.17024(17)	0.54309(7)	0.4951(3)	0.0232(9)
O4	0.17432(16)	0.50348(8)	0.6119(3)	0.0255(9)
O5	0.38624(18)	0.55740(8)	0.0767(4)	0.0265(9)
O6	0.4214(2)	0.53797(8)	0.2409(4)	0.0342(10)
O7	0.41795(17)	0.57348(8)	0.4367(3)	0.0256(9)
O8	0.52890(16)	0.52662(7)	1.0602(3)	0.0160(7)
H8	0.528(3)	0.5406(6)	1.106(3)	0.024(16)*
N1	0.5613(2)	0.58710(10)	0.9538(4)	0.0262(11)
N2	0.5104(2)	0.63017(10)	0.9872(4)	0.0284(12)
N3	0.3738(2)	0.70113(10)	1.4979(4)	0.0233(11)
N4	0.4124(2)	0.70875(9)	1.3175(4)	0.0274(11)
C1	0.4246(2)	0.52569(11)	0.8125(5)	0.0170(11)
C2	0.3821(2)	0.53272(10)	0.7036(4)	0.0150(11)
C3	0.3125(2)	0.52405(11)	0.6866(4)	0.0170(12)
H3	0.2899	0.5125	0.7425	0.020*
C4	0.2772(2)	0.53262(10)	0.5867(4)	0.0166(11)
C5	0.3116(3)	0.54957(11)	0.5031(5)	0.0213(12)
H5	0.2877	0.5556	0.4363	0.026*
C6	0.3810(3)	0.55751(11)	0.5187(5)	0.0196(12)
C7	0.4154(3)	0.54959(11)	0.6186(4)	0.0192(12)
H7	0.4617	0.5555	0.6299	0.023*
C8	0.2010(2)	0.52550(11)	0.5632(5)	0.0189(11)
C9	0.3793(3)	0.59226(12)	0.3652(5)	0.0224(13)
C10	0.3719(3)	0.58685(11)	0.2466(5)	0.0206(13)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C11	0.3350(3)	0.60777(12)	0.1804(5)	0.0270(13)
H11	0.3303	0.6050	0.1002	0.032*
C12	0.3056(3)	0.63232(13)	0.2310(6)	0.0360(16)
H12	0.2807	0.6457	0.1856	0.043*
C13	0.3135(3)	0.63686(13)	0.3484(6)	0.0431(18)
H13	0.2933	0.6533	0.3832	0.052*
C14	0.3513(3)	0.61719(13)	0.4165(6)	0.0367(15)
H14	0.3577	0.6207	0.4959	0.044*
C15	0.3950(3)	0.55903(12)	0.1853(5)	0.0234(13)
C16	0.5012(3)	0.60178(12)	0.9622(5)	0.0290(14)
H16	0.4574	0.5932	0.9518	0.035*
C17	0.6113(3)	0.60817(13)	0.9759(5)	0.0351(15)
H17	0.6594	0.6047	0.9772	0.042*
C18	0.5815(3)	0.63436(13)	0.9953(5)	0.0375(16)
H18	0.6045	0.6520	1.0111	0.045*
C19	0.4559(3)	0.65296(12)	0.9948(5)	0.0362(15)
H19A	0.4102	0.6436	0.9923	0.043*
H19B	0.4596	0.6657	0.9271	0.043*
C20	0.4610(3)	0.67151(12)	1.1050(5)	0.0354(15)
H20A	0.5071	0.6806	1.1091	0.042*
H20B	0.4554	0.6590	1.1732	0.042*
C21	0.4046(3)	0.69547(12)	1.1063(5)	0.0345(15)
H21A	0.4027	0.7045	1.0295	0.041*
H21B	0.3594	0.6863	1.1207	0.041*
C22	0.4159(3)	0.71943(13)	1.1971(5)	0.0367(16)
H22A	0.4614	0.7284	1.1841	0.044*
H22B	0.3805	0.7346	1.1863	0.044*
C23	0.3597(3)	0.71244(12)	1.3950(5)	0.0251(13)
H23	0.3178	0.7220	1.3773	0.030*
C24	0.4401(3)	0.68891(14)	1.4853(5)	0.0332(15)
H24	0.4643	0.6789	1.5437	0.040*
C25	0.4644(3)	0.69377(13)	1.3751(5)	0.0368(16)
H25	0.5077	0.6881	1.3449	0.044*
O9	0.5000	0.5000	0.4119(7)	0.078(2)
H9	0.514(4)	0.4861(10)	0.368(5)	0.117*

refined as riding atoms with $U_{iso}(H) = 1.2U_{eq}(C)$. The H atom of the hydroxyl O atom was located in a difference Fourier map and refined with the O8—H8 distance restrained to 0.82 Å. The hydrogen of water molecule was refined with the O9—H9 distance restrained to 0.85 Å.

Discussion

Over the past three decades, various syntheses of coordination polymers (CPs) have received remarkable attention because of their potential applications materials [3–12]. Organic ligands play a key role in the design of the targeted structures. Compared with the rigid aromatic multicarboxylates, the flexible ligands or semi-rigid ligands can form more complicated and unusual structures because their flexible coordination possibilities. 5-(2-carboxyphenoxy) isophthalate as a semi-rigid aromatic multicarboxylic ligand has been used in construction of CPs [13–16]. Herein, we reported a coordination polymer constructed from Co(II), 5-(2-carboxyphenoxy)isophthalate and 1,4-di(1*H*-imidazol-1-yl)butane.

As shown in the figure, the asymmetric unit of the title complex contains two unique Co(II) ions, one fully deprotonated 5-(2-carboxyphenoxy)isophthalate ligand, one 1,4-di(1*H*-imidazol-1-yl)butane ligand, one μ_3-OH group and one half of a water molecule. Co1 shows a distorted CoO_4N_2 coordination and is defined by three oxygen atoms from three different 5-(2-carboxyphenoxy)isophthalate ligands, two nitrogen atoms from two different 1,4-di(1*H*-imidazol-1-yl)butane ligands, and one oxygen atom from μ_3-OH group. Co2 shows a distorted CoO_6 coordination and is defined by four oxygen atoms from three different 5-(2-carboxyphenoxy)isophthalate ligands and two oxygen atoms from two μ_3-OH groups. The tetranuclear core $[Co_4(\mu_3\text{-}OH)_2]^{6+}$ propagate in all directions via bridging carboxylates and N atoms into a 3D framework. In the crystal structure, there exist O—H···O and O—H···N hydrogen bonds, which further consolidate the crystal packing.

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