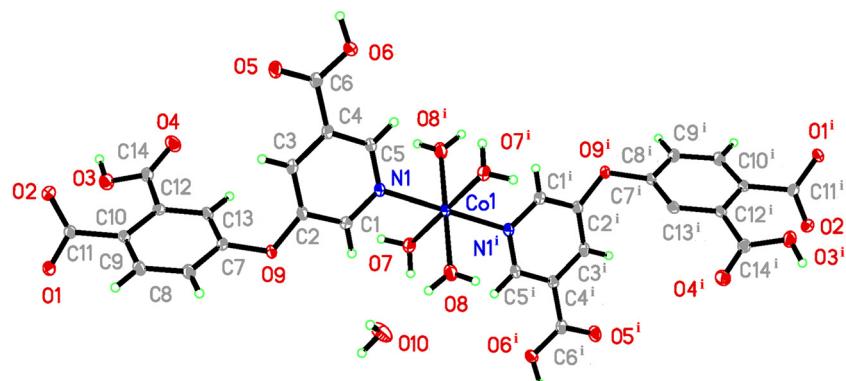


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Crystal structure of *trans*-tetraqua-bis{2-carboxy-4-((5-carboxypyridin-3-yl)oxy)benzoato- κ^1N }cobalt(II) dihydrate $C_{28}H_{28}O_{20}N_2Co$



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Abstract

$C_{28}H_{28}O_{20}N_2Co$, triclinic, $P\bar{1}$ (no. 2), $a = 7.7951(3)$ Å, $b = 9.3511(4)$ Å, $c = 11.3352(4)$ Å, $\beta = 105.499(4)^\circ$, $Z = 1$, $V = 762.19(6)$ Å 3 , $R_{gt}(F) = 0.0339$, $wR_{ref}(F^2) = 0.0798$, $T = 293.0$ K.

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The molecular structure is shown in Figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All chemicals were used without further purification. A mixture of $CoCl_2 \cdot 6H_2O$ (24.0 mg, 0.1 mmol), 4-(5-carboxy-

Table 1: Data collection and handling.

Crystal:	Pink block
Size:	0.36 × 0.32 × 0.29 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.66 mm $^{-1}$
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$,	10,420, 2842, 0.031
R_{int} :	
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2567
$N(param)_{refined}$:	244
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], Olex2 [4]

pyridine-3-yloxy)-phthalic acid (30.3 mg, 0.1 mmol) and 6 mL distilled water in a 20 mL Teflon-lined autoclave was kept under autogenous pressure at 353 K for three days. After cooling to room temperature at a rate of 5 K h $^{-1}$, pink crystals were collected by filtration and washed with distilled water.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3262 (3)	0.7465 (2)	0.63020 (19)	0.0254 (5)
H1	0.3471	0.8277	0.6889	0.031*
C2	0.2413 (3)	0.6054 (2)	0.65125 (18)	0.0220 (4)
C3	0.2094 (3)	0.4835 (2)	0.56603 (18)	0.0237 (4)
H3A	0.1517	0.3879	0.5787	0.028*
C4	0.2652 (3)	0.5068 (2)	0.46082 (18)	0.0217 (4)
C5	0.3488 (3)	0.6514 (2)	0.44464 (18)	0.0242 (5)
H5	0.3850	0.6665	0.3735	0.029*
C6	0.2345 (3)	0.3734 (2)	0.37034 (18)	0.0232 (4)
C7	0.2025 (3)	0.4783 (2)	0.82234 (17)	0.0218 (4)
C8	0.0676 (3)	0.4248 (2)	0.87936 (19)	0.0270 (5)
H8	-0.0342	0.4624	0.8681	0.032*
C9	0.0868 (3)	0.3141 (2)	0.9537 (2)	0.0273 (5)
H9	-0.0020	0.2793	0.9940	0.033*
C10	0.2347 (3)	0.2540 (2)	0.96931 (17)	0.0204 (4)
C11	0.2349 (3)	0.1250 (2)	1.04346 (18)	0.0216 (4)
C12	0.3685 (3)	0.3075 (2)	0.90871 (17)	0.0202 (4)
C13	0.3520 (3)	0.4218 (2)	0.83614 (18)	0.0225 (4)
H13	0.4419	0.4595	0.7973	0.027*
C14	0.5278 (3)	0.2461 (2)	0.91323 (18)	0.0242 (5)
Co1	0.5000	1.0000	0.5000	0.02458 (13)
N1	0.3795 (3)	0.77070 (19)	0.52806 (15)	0.0247 (4)
O1	0.2541 (2)	0.15340 (17)	1.15780 (13)	0.0334 (4)
O2	0.2086 (2)	-0.00127 (16)	0.98920 (13)	0.0271 (3)
O3	0.5723 (2)	0.17569 (19)	1.00879 (14)	0.0333 (4)
H3	0.6406	0.1263	0.9971	0.050*
O4	0.6086 (2)	0.2603 (2)	0.83498 (15)	0.0403 (4)
O5	0.1661 (2)	0.24835 (16)	0.39192 (14)	0.0346 (4)
O6	0.2861 (2)	0.39998 (18)	0.27250 (14)	0.0324 (4)
H6	0.264 (5)	0.308 (4)	0.220 (3)	0.095 (13)*
O7	0.6730 (2)	1.04588 (18)	0.68233 (14)	0.0361 (4)
H7A	0.6677	0.9648	0.7132	0.054*
H7B	0.6300	1.0962	0.7261	0.054*
O8	0.3075 (3)	1.0771 (2)	0.56010 (16)	0.0384 (4)
H8A	0.2525	1.0465	0.6132	0.058*
H8B	0.2419	1.1271	0.5185	0.058*
O9	0.1820 (2)	0.59623 (16)	0.75530 (13)	0.0282 (4)
O10	0.1419 (3)	0.9888 (2)	0.73233 (16)	0.0518 (5)
H10A	0.0442	0.9166	0.7021	0.078*
H10B	0.1606	0.9937	0.8100	0.078*

Comment

Coordination polymers, built from metal ions and organic linkers, have developed rapidly as important functional materials for their intriguing architectures and unique physical/chemical characteristics in gas storage [5], catalysis [6–8], chemical sensing [9, 10], luminescence devices [11, 12], and so on. At present, a variety of polycarboxylic acid ligands have been employed to construct a series of coordination polymers. In this work, we report a compound based on central Co(II) ions and 4-(5-carboxy-

pyridine-3-yloxy)-phthalic acid (H_3L) ligand under hydrothermal conditions. From the point of view of structural chemistry, H_3L is an efficient ligand, which contains three different types of functionalities ($-\text{COOH}$, N -pyridyl and O-ether) and has rich coordination modes. However, coordination polymers based on H_3L are still rare [13–16].

Single crystal X-ray structural analysis reveals that the asymmetric unit consists of one half of a Co(II) ion, one H_2L^- ligand, two coordinated water molecules and one lattice water molecule. The Co(II) ion exhibits a six-coordinated octahedral geometry, defined by four water O atoms (O_7 , O_8 , O_7^i and O_8^i) and two pyridyl N atoms (N_1 , and N_1^i). The distances of Co–O/N bond lengths (2.0693(16)–2.1872(17) Å) are in accordance with the normal ranges [17, 18], and the bond angles around Co atoms vary from 87.43(7)° to 180°. In the title complex, the carboxyl groups in H_2L^- are partly deprotonated, in which H_2L^- anions connect central Co(II) ion via pyridyl N atoms to form a discrete molecule structure. Moreover, there are hydrogen bonding interactions between lattice water molecules, coordinated water molecules and carboxyl oxygen atoms. These hydrogen bonding interactions contribute to the formation of a three-dimensional supramolecular architecture.

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