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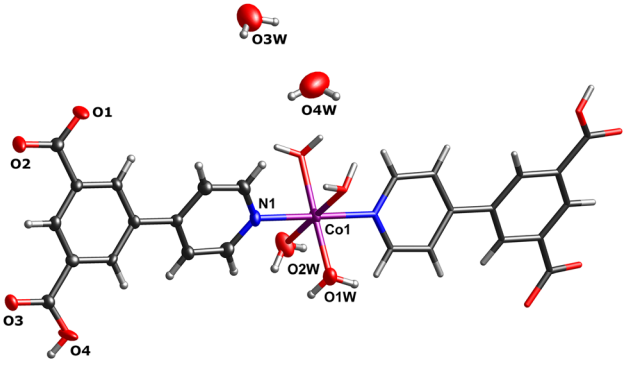
Crystal structure of [tetraaqua-bis((3-carboxy-5-(pyridin-4-yl)benzoate-κ^1N)cobalt(II)] tetrahydrate, $C_{26}H_{32}CoN_2O_{16}$



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Abstract

$C_{26}H_{32}CoN_2O_{16}$, orthorhombic, *Fdd*2 (no. 43), a = 24.8013(10) Å, b = 35.8890(16) Å, c = 6.9911(4) Å, V = 6222.7(5) Å^3, Z = 8, $R_{gt}(F)$ = 0.0487, $wR_{ref}(F^2)$ = 0.1462, T = 293(2) K.

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

1 Source of material

A mixture of $Co(NO_3)_2{\cdot}6H_2O$ (1 mmol, 291.0 mg), 5-(pyridin-4-yl)isophthalic acid (H_2pipa) (2 mmol, 486.4 mg), *N*,*N*-dimethylacetamide (1 mL) and H_2O (1.5 mL) were sealed in a 10 mL Teflon-lined autoclave that was heated at 358 K under autogenous pressure for two days. After cooling to room temperature, orange block crystals were obtained.

Table 1: Data collection and handling.

Crystal:	Orange block
Size:	0.20 × 0.18 × 0.16 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.63 mm^{-1}
Diffractometer, scan mode:	Multiwire proportional, φ and ω
θ_{max}, completeness:	25.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14,060, 2734, 0.044
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2530
$N(param)_{refined}$:	206
Programs:	Bruker [1], SHELX [2], PLATON [3]

2 Experimental details

The C-bound H atoms were placed in idealized positions (C—H = 0.93 Å) and refined by the riding model 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 distance restraint of O—H = 0.82 Å. The hydrogen of water molecule was refined with the distance restraint of O—H = 0.85 Å. The U_{iso} values were constrained to be 1.5U_{eq} of the carrier atom for oxygen H atoms.

3 Comment

As a rigid ligand containing one pyridyl and two carboxylic acid groups, 5-(pyridin-4-yl)isophthalic acid (H_2pipa) was widely used in the synthesis of multidimensional coordination polymers due to its excellent coordination ability and versatile coordination modes [4–11]. The two carboxylate groups can provide more coordination sites and the pyridyl group can complement the dicarboxylate ligands in the coordination and formation of high-connected nets. To further expand this research, 5-(pyridin-4-yl)isophthalic acid was selected as the organic ligand to synthesize a new Co(II) coordination polymer.

The asymmetric unit consists of half of a Co(II) center located on a crystallographic twofold axis, one partial

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.750000	0.750000	0.05215 (16)	0.0224 (3)
O1	1.08892 (18)	0.86268 (12)	0.1550 (9)	0.0483 (14)
O1W	0.75410 (16)	0.70957 (17)	−0.1606 (8)	0.0393 (14)
H1WA	0.722396	0.702653	−0.190217	0.059*
H1WB	0.771867	0.690993	−0.119268	0.059*
O2	1.16635 (17)	0.83449 (12)	0.2028 (9)	0.0449 (13)
O2W	0.75527 (17)	0.70830 (17)	0.2628 (8)	0.0442 (15)
H2WA	0.723779	0.700858	0.291862	0.066*
H2WB	0.773529	0.690099	0.219702	0.066*
O3	1.18028 (14)	0.69845 (11)	0.0478 (7)	0.0293 (9)
O4	1.10550 (17)	0.66870 (12)	−0.0287 (10)	0.0477 (15)
H4	1.123956	0.650184	−0.007945	0.072*
N1	0.83629 (16)	0.75392 (14)	0.0545 (11)	0.0244 (10)
C1	0.8619 (2)	0.78621 (17)	0.0294 (11)	0.0283 (14)
H1	0.841224	0.807527	0.010883	0.034*
C2	0.9179 (2)	0.78992 (17)	0.0293 (10)	0.0269 (13)
H2	0.933795	0.813003	0.007410	0.032*
C3	0.9497 (2)	0.75855 (14)	0.0623 (9)	0.0193 (12)
C4	0.9231 (2)	0.72458 (17)	0.0923 (8)	0.0243 (14)
H4A	0.942756	0.702928	0.114133	0.029*
C5	0.8675 (2)	0.72358 (17)	0.0890 (9)	0.0258 (15)
H5	0.850508	0.700914	0.111680	0.031*
C6	1.0096 (2)	0.76110 (15)	0.0691 (8)	0.0200 (12)
C7	1.0348 (2)	0.79508 (16)	0.1064 (8)	0.0221 (12)
H7	1.014104	0.816342	0.125772	0.026*
C8	1.0911 (2)	0.79736 (15)	0.1148 (8)	0.0214 (12)
C9	1.1220(2)	0.76569 (17)	0.0873 (8)	0.0225 (13)
H9	1.159396	0.767102	0.093122	0.027*
C10	1.0970 (2)	0.73183 (15)	0.0511 (9)	0.0200 (11)
C11	1.0413 (2)	0.72978 (15)	0.0399 (9)	0.0188 (11)
H11	1.024930	0.707085	0.012490	0.023*
C12	1.1312 (2)	0.69771 (16)	0.0229 (9)	0.0237 (13)
C13	1.1181 (2)	0.83389 (16)	0.1619 (10)	0.0263 (13)
O3W	0.7593 (3)	0.8855 (2)	0.7532 (14)	0.086 (2)
H3WA	0.726559	0.881426	0.724066	0.129*
H3WB	0.779639	0.877576	0.664208	0.129*
O4W	0.7532 (3)	0.8662 (3)	0.1527 (17)	0.117 (4)
H4WA	0.785335	0.858078	0.147490	0.141*
H4WB	0.737596	0.862317	0.046140	0.176*

deprotonated H_2pipa ligand, two coordinated water molecules and two free water molecules (see the figure). The Co(II) center has a distorted octahedral coordination geometry, which is connected to four oxygen atoms from two pairs of symmetry related water molecules (Co—O = 2.081(6)—2.104(6) Å), and two nitrogen atoms from two symmetry related Hpipa$^-$ ligands (Co—N = 2.145(4) Å). The carboxylic acid groups in the Hpipa$^-$ ligand do not participate in the coordination, and thus form a zero-dimensional structure. In the crystal structure, O—H···O hydrogen bonds link the tile molecules into a three dimensional supermolecular framework.

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