Min-Le Han*, Xiao-Dong Li and Yan-Hong Li

Crystal structure of poly[diaqua-(μ_3 -3,4,5,6-tetrafluoro-phthalato- κ^3 O:O':O'')-(μ_2 -1,2-bis(4-pyridyl)ethene- κ^2 N:N')cobalt(II)], $C_{14}H_9CoF_4NO_6$

Table 1: Data collection and handling.

Crystal:	Block, clear light pink
Size:	0.35 × 0.32 × 0.28 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	9.74 mm ⁻¹
Diffractometer, scan mode:	XtaLAB Pro, ω -scans
$\theta_{\max}$, completeness:	66.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7102, 2663, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2385
$N(\text{param})_{\text{refined}}$:	244
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3]

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Abstract

$C_{14}H_9CoF_4NO_6$, monoclinic, $P2_1/n$ (no. 14), $a = 6.1183(2)$ Å, $b = 7.5688(2)$ Å, $c = 32.5016(7)$ Å, $\beta = 90.672(2)^\circ$, $V = 1504.99(7)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0398$, $wR_{\text{ref}}(F^2) = 0.1037$, $T = 293$ K.

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A part of the title crystal 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.

Source of material

A mixture of 3,4,5,6-tetrafluoro-phthalic acid ($H_2F_4\text{ph}$, 24 mg, 0.1 mmol), 1,2-bis(4-pyridyl)ethene (18 mg, 0.1 mmol), and

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.37240(8)	0.35340(6)	0.29419(2)	0.02168(16)
F1	−0.0368(4)	0.6877(3)	0.37152(6)	0.0443(5)
F2	0.1114(5)	0.7922(3)	0.44562(6)	0.0604(7)
F3	0.4991(5)	0.9662(3)	0.45256(6)	0.0643(8)
F4	0.7238(4)	1.0521(3)	0.38459(6)	0.0478(6)
O1	0.1779(4)	0.5852(3)	0.29129(7)	0.0302(5)
O2	0.1529(4)	0.8636(3)	0.27003(6)	0.0260(5)
O3	0.6625(4)	0.8567(3)	0.28218(8)	0.0370(6)
O4	0.5858(4)	1.1356(3)	0.29546(7)	0.0303(5)
O5	0.6614(4)	0.4968(3)	0.28730(6)	0.0284(5)
H5A	0.6336	0.6085	0.2856	0.043*
H5B	0.7197	0.4725	0.2639	0.043*
O6	0.0875(4)	0.1994(3)	0.29813(7)	0.0299(5)
N1	0.3860(4)	0.3744(3)	0.35996(7)	0.0250(6)
C1	0.1909(5)	0.7463(4)	0.29657(9)	0.0225(6)
C2	0.5782(5)	0.9746(4)	0.30318(9)	0.0245(7)
C3	0.2673(5)	0.8149(4)	0.33843(9)	0.0243(7)
C4	0.1536(5)	0.7761(4)	0.37358(9)	0.0292(7)
C5	0.2277(7)	0.8304(5)	0.41182(10)	0.0397(9)
C6	0.4223(7)	0.9201(5)	0.41535(10)	0.0391(9)
C7	0.5339(6)	0.9633(4)	0.38022(10)	0.0316(7)
C8	0.4576(5)	0.9164(4)	0.34157(9)	0.0229(6)
C9	0.5657(6)	0.4426(5)	0.37774(9)	0.0330(8)
H9	0.6824	0.4727	0.3611	0.040*
C10	0.5863(6)	0.4704(5)	0.41932(10)	0.0367(8)
H10	0.7134	0.5206	0.4301	0.044*
C11	0.4170(6)	0.4235(5)	0.44534(9)	0.0338(8)
C12	0.2330(6)	0.3503(5)	0.42707(10)	0.0362(8)

*Corresponding author: Min-Le Han, College of Chemistry and Chemical Engineering, Henan Key Laboratory of Function-Oriented Porous Materials, Luoyang Normal University, Luoyang, Henan 471934, P.R. China, e-mail: minle_han@163.com. <https://orcid.org/0000-0003-3934-3345>

Xiao-Dong Li and Yan-Hong Li: College of Chemistry and Chemical Engineering, Henan Key Laboratory of Function-Oriented Porous Materials, Luoyang Normal University, Luoyang, Henan 471934, P.R. China

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H12	0.1161	0.3155	0.4432	0.043*
C13	0.2225(6)	0.3286(5)	0.38472(10)	0.0321(8)
H13	0.0965	0.2802	0.3731	0.039*
C14	0.4261(7)	0.4523(5)	0.49001(10)	0.0415(9)
H14	0.3183	0.3987	0.5056	0.050*
H6A	0.118(8)	0.096(3)	0.2886(13)	0.056(13)*
H6B	−0.035(5)	0.240(7)	0.2904(16)	0.088(19)*

Co(OAc)₂·4H₂O (24 mg, 0.1 mmol) was added to water (7 mL) in a 25 mL Teflon-lined autoclave. The mixture was heated at 393 K for 3 days and then slowly cooled to room temperature. Light pink block crystals were obtained.

Experimental details

The carbon bound hydrogen atoms were placed in calculated positions and refined using a riding model on attached atoms.

Discussion

In the last two decades, a large number of Co(II)-metal-organic frameworks (MOFs) have been reported in the literature due to their potential applications in many areas [4–8]. Co(II)-MOFs can be constructed from Co(II) ions and carboxylate and nitrogen-containing ligands. In most situations, *N*-containing ligands are electrically neutral, while carboxylate ligands are negatively charged. Among rod *N*-donor ligands, 1,2-bis(4-pyridyl)ethene (bpe) can serve as a bridging ligand [9–11]. On the other hand, phthalic acid (1,2-benzenedicarboxylic acid) and its derivatives, can yield predetermined networks [12, 13].

In the title complex, there are one Co(II) ion, two coordinated water molecules, one F₄ph ligand, and half of bpe ligand in the asymmetric unit (Figure). Co1 is six-coordinated with a distorted octahedral geometry by three O atoms from three F₄ph ligands and one N atom of one bpe ligand, and two coordinated water molecules with an O5–Co1–O6'' angle of 176.53(9)°. The Co–N bond length is 2.144(2) Å and the Co–O ones are in the range of 2.089(2) and 2.121(2) Å. Each F₄ph ligand acts as a μ₃-bridge, in which one carboxylate group adopts a bi-monodentate bridging mode to link two cobalt(II) atoms while another adopts a monodentate mode (see the figure). Co(II) ions are linked by μ₂-bpe ligands and μ₃-F₄ph ligands to form a 2-D layer. In the title complex, there are several hydrogen bonds: hydrogen bonding between water and carboxylate O atoms with d(O···O) = 2.729(3), 2.732(3) Å and 2.728(3) Å. The 2-D layers are further linked by hydrogen bonds to generate a 3-D architecture.

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