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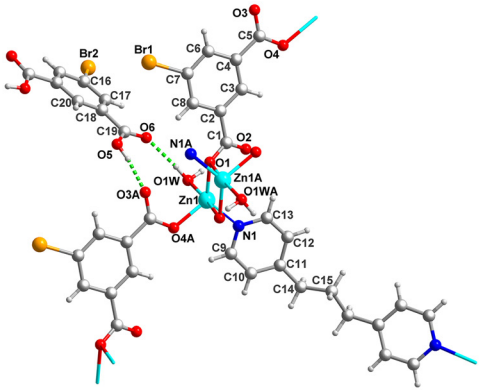
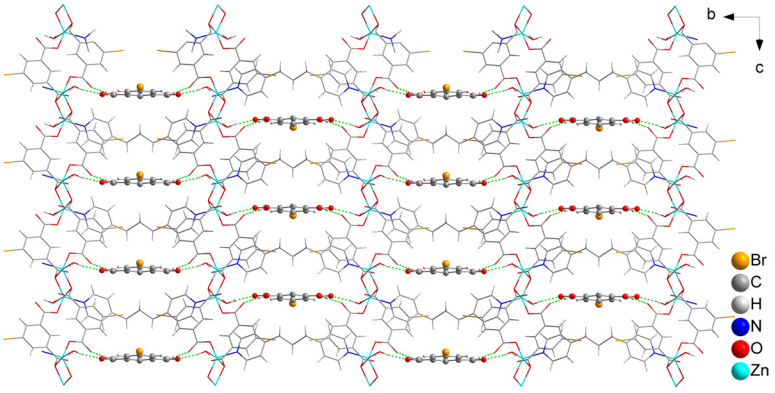
The crystal structure of poly[diaqua-bis(μ_3-5-bromobenzene-1,3-dicarboxylato-κ^3O,O,O')-(μ_2-1,3-bis-(4-pyridyl)-propane-κ^2N,N')-dizinc(II))] – 5-bromobenzene-1,3-dicarboxylic acid [2/1], $C_{37}H_{29}Br_3N_2O_{14}Zn_2$





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Abstract

$C_{37}H_{29}Br_3N_2O_{14}Zn_2$, monoclinic, $P2_1/m$ (no. 11), a = 8.2047(6) Å, b = 28.6129(13) Å, c = 8.8721(7) Å, β = 112.069(9)°, V = 1930.2(3) Å^3, Z = 2, $R_{gt}(F)$ = 0.0588, w$R_{ref}(F^2)$ = 0.1547, T = 200 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.15 × 0.10 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	5.80 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	70.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7278, 3722, 0.038
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3196
$N(param)_{refined}$:	270
Programs:	CrysAlis^{PRO} [1], Olex2 [2, 4], SHELX [3]

Source of materials

A mixture of $ZnCl_2$ (0.136 g, 1 mmol), 5-bromobenzene-1,3-dicarboxylic acid (0.245 g, 1 mmol), and 1,3–bis-(4-pyridyl)-propane (0.198 g, 1 mmol) was added to 10 mL H_2O, stirred to form a clear solution. The mixture was heated in an oven at 413 K for 72 h. Many colourless crystals were filtered, washed with distilled water, and dried in air. Yield: 36.8% (based on 5-bromobenzene-1,3-dicarboxylic acid).

Experimental details

The structure was solved by Direct Methods with the SHELXS-2018 program. All H-atoms from C and N atoms were positioned with idealized geometry and refined isotropically

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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}
Br1	0.84316 (10)	0.69306 (2)	0.86182 (11)	0.0694 (3)
C1	0.8177 (6)	0.51884 (16)	0.6454 (5)	0.0223 (9)
C2	0.9085 (6)	0.55394 (16)	0.7757 (5)	0.0205 (9)
C3	1.0623 (6)	0.54196 (16)	0.9045 (5)	0.0215 (9)
H3	1.109004	0.512098	0.910177	0.026*
C4	1.1466 (6)	0.57453 (16)	1.0249 (6)	0.0212 (9)
C5	1.3124 (6)	0.56120 (16)	1.1662 (5)	0.0230 (9)
C6	1.0800 (7)	0.61983 (17)	1.0140 (6)	0.0285 (10)
H6	1.135485	0.642027	1.093320	0.034*
C7	0.9302 (7)	0.63073 (17)	0.8830 (7)	0.0316 (11)
C8	0.8397 (6)	0.59861 (17)	0.7643 (6)	0.0278 (10)
H8	0.735892	0.606778	0.679483	0.033*
C9	0.5000 (7)	0.41848 (18)	0.1260 (6)	0.0302 (11)
H9	0.422901	0.440187	0.057764	0.036*
C10	0.5047 (8)	0.37412 (18)	0.0685 (6)	0.0339 (12)
H10	0.429554	0.366406	−0.036433	0.041*
C11	0.6188 (7)	0.34085 (17)	0.1635 (6)	0.0298 (11)
C12	0.7234 (9)	0.3547 (2)	0.3177 (7)	0.0505 (17)
H12	0.801700	0.333513	0.387550	0.061*
C13	0.7140 (8)	0.39959 (19)	0.3705 (7)	0.0419 (14)
H13	0.787745	0.408003	0.475253	0.050*
C14	0.6304 (9)	0.29337 (18)	0.0931 (7)	0.0404 (14)
H14A	0.737611	0.292644	0.071240	0.049*
H14B	0.532903	0.290757	−0.010772	0.049*
C15	0.6288 (10)	0.250000	0.1927 (9)	0.0301 (15)
H15A	0.524352	0.250000	0.219200	0.036*
H15B	0.731169	0.250001	0.293470	0.036*
N1	0.6022 (5)	0.43153 (14)	0.2764 (5)	0.0247 (8)
O1	0.6554 (4)	0.52702 (12)	0.5594 (4)	0.0251 (7)
O1W	0.7523 (4)	0.53360 (12)	0.2696 (4)	0.0263 (7)
H1WA	0.853929	0.521549	0.314902	0.040*
H1WB	0.763906	0.561399	0.305998	0.040*
O2	0.8997 (4)	0.48513 (12)	0.6233 (4)	0.0286 (7)
O3	1.3820 (5)	0.58995 (13)	1.2773 (4)	0.0350 (8)
O4	1.3671 (4)	0.52045 (12)	1.1609 (4)	0.0257 (7)
Zn1	0.57427 (8)	0.49777 (2)	0.34306 (7)	0.02328 (19)
Br2	1.24553 (12)	0.750000	0.27401 (13)	0.0516 (3)
C16	1.0350 (9)	0.750000	0.3139 (9)	0.0296 (15)
C17	0.9613 (6)	0.70797 (17)	0.3290 (6)	0.0279 (10)
H17	1.013916	0.679909	0.319593	0.033*
C18	0.8063 (6)	0.70828 (16)	0.3587 (6)	0.0260 (10)
C19	0.7228 (7)	0.66176 (17)	0.3618 (6)	0.0282 (10)
C20	0.7280 (9)	0.750000	0.3752 (9)	0.0266 (14)
H20	0.625498	0.750000	0.396785	0.032*
O5	0.5639 (5)	0.66493 (13)	0.3609 (6)	0.0443 (10)
H5	0.515651	0.639341	0.339307	0.066*
O6	0.7973 (5)	0.62577 (13)	0.3585 (6)	0.0440 (10)

(U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O)) using a riding model with C—H = 0.93 and 0.97 Å, and O—H = 0.850 and 0.851 Å, respectively. The bromine atom is slightly disordered.

Comment

Zinc(II) coordination polymers with 5-substituted isophthalic acid such as 5-hydroxy [5], 5-methoxy [6], 5-methyl [6, 7], 5-sulfo [8], 5-nitro [9–11], 5-azido [12] and other substituents show a variety of topological structures [13–16]. The flexible 1,3–bis-(4-pyridyl)-propane with a longer linker between two pyridyl groups is used as a secondary building block in the carboxylate systems and may show different configuration when coordinated to metal ions.

As shown in the left part of the figure, the asymmetric unit consists of one Zn(II) cation, one completely deprotonated 5-bromo-1,3-dicarboxylate anion, a half 1,3–bis-(4-pyridyl)-propane, one coordinated water molecule, and one half of a neutral uncoordinated 5-bromo-1,3-dicarboxylic acid. The Zn(II) is coordinated by four atoms (N1, O1, O4, and O1W) from one pyridyl nitrogen atom of 1,3–bis-(4-pyridyl)-propane ligand, two oxygen atoms of two different bridging 5-bromo-1,3-dicarboxylate anions and one water molecule to form a highly distorted tetrahedral configuration.

The bond distance of Zn1–N1 (2.024(4) Å) is typical for Zn–N_{py} coordination. The bond distance between monodentate carboxylate oxygen atom and Zn(II) [1.964(3) Å] is shorter than those between chelating carboxylate oxygen atoms and Zn(II) [1.967(3) and 2.455(3) Å]. Every two neighbouring Zn(II) cations are linked by O1 atoms to form dimers, which are further connected by 1,3–bis-(4-pyridyl)-propane and 5-bromo-1,3-dicarboxylate to generate one 2D porous structure. The uncoordinated 5-bromo-1,3-dicarboxylic acid molecules are anchored in the pores by two kinds of strong hydrogen bonds, O1W—H1B···O6 and O5—H5···O3 with the distances of H atoms and acceptors 1.894 and 1.746 Å. A 3D structure is obtained thorough these 2D layers, which are linked by strong hydrogen bond O1W—H1A···O2 with the distance of H atom and acceptor 1.894 Å demonstrated in the right part of the figure. All the bond lengths of the title compound are similar to the reported results [17].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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