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Crystal structure of poly[aqua-(μ_2 -1,4-bis(2'-carboxylatophenoxy)benzene- $\kappa^2 O:O'$)-(μ_2 -4,4'-bipyridione- $\kappa^2 N:N'$)cadmium(II)] monohydrate, $C_{30}H_{22}CdN_2O_7 \cdot H_2O$

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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:	Colorless plate
Size:	0.30 × 0.25 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.84 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, ω
$\theta_{\max}$, completeness:	25.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6385, 2586, 0.047
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 1834
$N(\text{param})_{\text{refined}}$:	200
Programs:	Bruker [1], SHELX [2–4]

Source of material

All chemicals were of analytical reagent grade and used without further purification. A mixture of $Cd(NO_3)_2 \cdot 4H_2O$ (30.8 mg, 0.1 mmol), H_2bcpb (17.5 mg, 0.05 mmol), 4,4'-bipy (15.6 mg, 0.1 mmol), H_2O (6.0 mL) and NaOH (0.5 mL 0.25 mol/L) was placed in a 25 mL of Teflon-lined stainless steel vessel, which was heated at 140 °C for three days. Followed by slow cooling to room temperature, colorless plate crystals of title compound were obtained in 55.7% yield.

Experimental details

Multi-scan program SADABS [1] was used for absorption correction. The structures were solved by direct methods and refined using SHELXS-97 [2, 3] and SHELXL-2014/7 [4]. Hydrogen atoms attached to C atoms were placed geometrically and refined using a riding model approximation, with C–H = 0.96 Å and $U_{\text{iso}}(H) = 1.2 U_{\text{eq}}(C)$.

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Abstract

$C_{30}H_{24}CdN_2O_8$, orthorhombic, *Pccn* (no. 56), $a = 11.720(2)$ Å, $b = 14.000(3)$ Å, $c = 16.971(3)$ Å, $V = 2784.7(10)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0544$, $\omega R_{\text{ref}}(F^2) = 0.1664$, $T = 293(2)$ K.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
Cd1	0.2500	0.7500	0.29086 (3)	0.0461 (2)
O1	0.2441 (3)	0.6338 (3)	0.3837 (4)	0.0712 (14)
O2	0.2364 (5)	0.5334 (7)	0.2883 (3)	0.121 (3)
O3	0.2694 (3)	0.5282 (3)	0.5278 (2)	0.0545 (10)
O4 ^a	0.2514 (8)	0.6683 (10)	0.1768 (6)	0.090 (3)
H4A ^a	0.234 (14)	0.689 (5)	0.1316 (19)	0.135*
H4B ^a	0.278 (15)	0.612 (5)	0.177 (3)	0.135*
N1	0.4479 (5)	0.7490 (3)	0.2892 (3)	0.0535 (13)
C1	0.5075 (5)	0.8231 (5)	0.2596 (4)	0.0607 (16)
H1	0.4674	0.8748	0.2390	0.073*
C2	0.6260 (4)	0.8259 (5)	0.2583 (4)	0.0614 (17)
H2	0.6639	0.8785	0.2374	0.074*
C3	0.6253 (4)	0.6734 (5)	0.3183 (4)	0.0574 (15)
H3	0.6632	0.6204	0.3384	0.069*
C5	0.5079 (4)	0.6763 (5)	0.3184 (3)	0.0570 (15)
H5	0.4684	0.6249	0.3398	0.068*
C6	0.2220 (5)	0.5533 (5)	0.3563 (4)	0.0587 (15)
C7	0.1672 (4)	0.4802 (4)	0.4101 (3)	0.0487 (12)
C8	0.0833 (5)	0.4219 (5)	0.3767 (4)	0.0657 (17)
H8	0.0691	0.4256	0.3229	0.079*
C9	0.0215 (6)	0.3592 (5)	0.4223 (6)	0.079 (2)
H9	−0.0351	0.3217	0.3995	0.095*
C10	0.0433 (6)	0.3523 (5)	0.5000 (6)	0.089 (3)
H10	0.0017	0.3096	0.5306	0.106*
C11	0.1265 (6)	0.4079 (6)	0.5347 (4)	0.075 (2)
H11	0.1410	0.4028	0.5884	0.091*
C12	0.1886 (4)	0.4716 (4)	0.4887 (3)	0.0502 (13)
C13	0.3845 (4)	0.5112 (4)	0.5111 (3)	0.0465 (12)
C14	0.4242 (4)	0.4313 (4)	0.4724 (3)	0.0543 (14)
H14	0.3736	0.3850	0.4545	0.065*
C15	0.4595 (4)	0.5791 (4)	0.5394 (3)	0.0551 (15)
H15	0.4318	0.6320	0.5664	0.066*
C4	0.6868 (5)	0.7495 (5)	0.2883 (3)	0.0520 (15)
O5	0.2500	0.7500	0.5239 (4)	0.069 (2)
H5A	0.255 (7)	0.791 (5)	0.487 (4)	0.104*

^aOccupancy: 0.5.

Hydrogen atoms attached to O atoms were located from difference Fourier maps and refined using $U_{iso}(H) = 1.5 U_{eq}(O)$.

Comment

In the past few decades, ether bond polycarboxylic coordination polymers (CPs) with structural and functional tenability [5] have attracted considerable attention from both academia and industry on account of their exploitable properties for potential application in areas such as separation [6], catalysis [7], ion exchange [8, 9], chirality, magnetism [10] luminescence [11], and nonlinear optics. We recently studied the self-assembly reaction of

1,4-bis(2'-carboxylatophenoxy)benzene (H_2bcpb) and metal ions. Herein, we report a cadmium(II) compound with two-dimensional structure, namely $[Cd(\mu_2-bcpb)(4,4-bipy)(H_2O), H_2O]_n$ ($bcpb^{2-}$ = deprotonated 1,4-bis(2'-carboxylatophenoxy)benzene; 4,4-bipy = 4,4-bipyridine).

Single-crystal X-ray analysis shows that the compound crystallizes in the orthorhombic space group $Pccn$ with a 2D structure. The asymmetric unit contains one half of a Cd(II) cation, one half of a $bcpb^{2-}$ anion, one half of a 4,4-bipy, one half of a coordinated water molecule and one half of a free water molecule. The Cd1 atom is six-coordinated and forms a slightly distorted octahedron geometry, which is completed by two carboxylate O donors coming from two $bcpb^{2-}$ moieties, two N donors from 4,4-bipy ligand and two O atoms from two water molecules. The bond lengths of the Cd–O bonds are in the range of 2.247(10)–2.266(5) Å, and the distance of the Cd–N bond is 2.320(6) Å. The O–Cd–O angles are within the range of 61.2(7)–164.5(4)°. The $bcpb^{2-}$ ligand connects two Cd(II) ions with a $\mu_2-k^2O^1:O^1$ -coordination mode. In the crystal, intermolecular O–H...O hydrogen bonds involving the carboxylate oxygen atoms and lattice water molecules link the moieties into a three-dimensional network. The free water molecule and $bcpb^{2-}$ ligand were linked together through $O5-H5A...O3^{ii}$ and $O5-H5A...O1^{ii}$ (Symmetry codes: (ii) $-x + 1/2, -y + 3/2, z$) hydrogen bonds. Additionally, short $\pi \cdots \pi$ interactions between Cg1 and Cg1ⁱ occur in the structure (Symmetry codes: (i) $-x, 1 - y, 1 - z$). Cg1 ring is composed of C7–C12. The geometry analysis shows a face-to-face alignment of the aromatic-aromatic rings. The distance between Cg1 ring and Cg1^v ring is 3.738(4) Å.

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