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Crystal structure of *catena*-poly[octaaqua-bis(μ_2 -4,6-dicarboxyisophthalate- $\kappa^2 O:O'$)cadmium(II) disodium(I)] dihydrate, $C_{20}H_{28}CdNa_2O_{26}$

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Abstract

$C_{20}H_{28}CdNa_2O_{26}$, monoclinic, $C2/m$ (no. 12), $a = 7.4169(8)$ Å, $b = 20.183(2)$ Å, $c = 10.5403(11)$ Å, $\beta = 103.5520(10)^\circ$, $V = 1533.9(3)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0434$, $wR_{ref}(F^2) = 0.1271$, $T = 296(2)$ K.

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A part of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Prism, colorless
Size:	$0.20 \times 0.05 \times 0.05$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.85 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
θ_{max} , completeness:	28.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	9620, 1988, 0.044
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1714
$N(param)_{refined}$:	121
Programs:	Bruker programs [1], SHELX [2]

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

Atom	x	y	z	U_{iso}^*/U_{eq}
Cd1	0.0000	0.0000	0.0000	0.03157(19)
Na1	0.3202(3)	0.0000	0.33009(18)	0.0328(4)
C1	0.5000	0.1870(2)	0.5000	0.0212(8)
H1	0.5000	0.1409	0.5000	0.025*
C2	0.5000	0.3211(2)	0.5000	0.0202(8)
H2	0.5000	0.3672	0.5000	0.024*
C3	0.3971(4)	0.21918(13)	0.3901(3)	0.0187(6)
C4	0.3970(4)	0.28891(14)	0.3900(3)	0.0182(5)
C5	0.2960(4)	0.33625(15)	0.2837(3)	0.0230(6)
C6	0.2959(4)	0.17132(15)	0.2848(3)	0.0263(6)
O1	0.3071(3)	0.39580(11)	0.3048(2)	0.0324(5)
O2	0.2042(4)	0.31277(13)	0.1771(2)	0.0483(8)
O3	0.2040(5)	0.19466(12)	0.1763(3)	0.0523(9)
O4	0.3040(4)	0.11283(12)	0.3051(3)	0.0443(7)
O5	0.6633(6)	0.0000	0.4355(4)	0.0433(9)
O6	0.3102(5)	0.0000	0.0920(3)	0.0302(7)
O7	-0.0239(5)	0.0000	0.2123(3)	0.0339(8)
O8	0.0000	0.10786(16)	0.0000	0.0470(11)
O9	0.5000	0.09711(15)	0.0000	0.0294(7)
H8	0.0515	0.1361	0.0443	0.035*
H5	0.7021	-0.0310	0.3984	0.035*
H9	0.4477	0.1264	-0.0461	0.035*
H6	0.3498	0.0317	0.0665	0.035*
H7	-0.0671	-0.0326	0.2410	0.035*
H3	0.1981	0.2450	0.1745	0.035*

Source of material

A mixture of benzene-1,2,4,5-tetracarboxylic acid (H_4Btec ; 0.05 g), $CdSO_4$ (0.05 g), $NaOH$ (0.03 g) and deionized water (10 mL) was placed in a 25-mL Teflon-lined stainless steel

autoclave. Then the autoclave was heated to 160 °C under autogenous pressure for 120 h. After being slowly cooled to room temperature at a rate of 5 °C/h, colorless prismatic crystals were recovered by filtration, washed with distilled water and air dried.

Experimental details

The C-bound H atom was positioned geometrically and refined with a riding model: C—H = 0.93 Å with $U_{\text{iso}}(\text{H}) = 1.2$. The water H atoms were located in the difference fourier maps, and then refined with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{O})$.

Discussion

A considerable amount of experimental data has been accumulated for the purpose of developing new coordination polymers with fascinating topology and interesting physical properties [3, 4]. Crystal engineering provides a powerful tool for the design and synthesis of novel compounds, and the advances in theory and practice of molecular crystallography have increased the speed and scope of structure determination enormously. Organic aromatic polycarboxylate ligands, as one kind of multi-dentate O-donor ligands have been extensively employed in the preparation of coordination polymer in possession of multi-dimensional networks and interesting properties [5, 6]. Taking these advantages into account, recently we have begun to assemble H₄btec, sodium ions and cadmium ions into polymeric complexes under hydrothermal conditions.

Single crystal X-ray analysis shows that the title compound exhibits a one-dimensional chain. This structure is isostructural to other reported compounds that the Cd²⁺ ions were substituted by Co²⁺ [7], Zn²⁺ [8], Mn²⁺, Ni²⁺ [9], Cu²⁺ [10] and Mg²⁺ [11]. There is one quarter of a Cd^{II} atom, one half of a Na^I atom, one half H₂Btec ligand, four halves coordinating aqua and one half of a lattice water in the asymmetric unit. Each Na atom is hexa-coordinated by O atoms including two carboxylate O atoms and four water O atoms for a distorted NaO₆ octahedron with the Na—O bond distances ranging from 2.292(2) to 2.563(4) Å. The Cd atom also resides in a CdO₆ octahedron with the Cd—O bond distances ranging from 2.177(3) to 2.286(3) Å. The CdO₆ octahedra connect NaO₆ octahedra via edge-sharing O atoms to form a chain of [—NaO₆—CdO₆—NaO₆—]_∞, whose two sides are occupied by half deprotonated H₂Btec ligands. There exists two intramolecular hydrogen bonds in each H₂Btec ligand with the two O—H distances, O2—H3 = 1.37 Å and O3—H3 = 1.02 Å.

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