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Crystal structure of coordination polymer *catena*-poly[*diaqua*-(2,6-dichloropyridine-4-carboxylato- $\kappa^2 O, O'$)-bis(μ_2 -2,6-dichloropyridine-4-carboxylato- $\kappa^2 O:O'$)]terbium(III), $C_{18}H_{10}Cl_6N_3O_8Tb$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.37 × 0.32 × 0.31 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	3.56 mm ⁻¹
Diffractometer, scan mode:	SuperNova EosS2, ω
$\theta_{\max}$, completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9301, 2297, 0.029
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2240
$N(\text{param})_{\text{refined}}$:	167
Programs:	Olex2 [1], SHELX [2, 3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Tb1	0.2500	0.52749(2)	0.5000	0.01547(8)
Cl1	0.14863(11)	0.44248(9)	0.20766(5)	0.0576(3)
Cl2	0.59081(13)	0.21827(11)	0.18615(5)	0.0708(4)
Cl3	0.09230(14)	−0.09182(8)	0.40163(5)	0.0756(4)
O1	0.4140(2)	0.47718(17)	0.43006(10)	0.0296(5)
O1W	0.3774(2)	0.69425(16)	0.47102(10)	0.0317(5)
H1WA	0.3327	0.7550	0.4625	0.048*
H1WB	0.4402	0.6760	0.4454	0.048*
O2	0.6249(2)	0.42622(17)	0.40830(9)	0.0277(5)
O3	0.1639(2)	0.33962(15)	0.46581(9)	0.0273(5)
N1	0.3765(3)	0.3355(2)	0.20797(12)	0.0355(6)
N2	0.2500	−0.0719(3)	0.5000	0.0474(12)
C1	0.5001(3)	0.4360(2)	0.39500(12)	0.0194(6)
C2	0.4541(3)	0.3961(2)	0.33016(12)	0.0204(6)
C3	0.3295(3)	0.4305(2)	0.30497(13)	0.0271(6)
H3	0.2692	0.4732	0.3283	0.032*
C4	0.2994(3)	0.3981(3)	0.24373(14)	0.0310(7)
C5	0.4924(4)	0.3019(3)	0.23419(15)	0.0353(8)
C6	0.5378(3)	0.3296(2)	0.29453(14)	0.0292(7)
H6	0.6212	0.3042	0.3104	0.035*
C7	0.2500	0.2883(3)	0.5000	0.0216(8)
C8	0.2500	0.1618(3)	0.5000	0.0233(8)
C9	0.1792(3)	0.1036(2)	0.45341(14)	0.0313(7)
H9	0.1315	0.1409	0.4213	0.038*
C10	0.1824(4)	−0.0121(3)	0.45657(17)	0.0409(9)

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Abstract

$C_{18}H_{10}Cl_6N_3O_8Tb$, monoclinic, $I2/a$ (no. 15), $a = 9.8281(2)$ Å, $b = 11.8792(2)$ Å, $c = 21.1568(4)$ Å, $\beta = 91.1599(19)^\circ$, $V = 2469.54(9)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0196$, $wR_{\text{ref}}(F^2) = 0.0473$, $T = 293(2)$ K.

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The crystal structure is shown in the figure (asymmetric unit shown as ellipsoids). Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

All reagents used in these syntheses were of analytical grade and used as purchased without further purification. The mixtures of 2,6-dichloropyridine-4-carboxylic acid (dcp)

(0.1 mmol, 19.2 mg), $Tb(NO_3)_3 \cdot 6H_2O$ (0.05 mmol, 22.7 mg), and H_2O (6.0 mL) were placed in a 23 mL Teflon liner stainless steel reactor. The vessel was heated to 433 K for 4 days, and then slowly cooled to room temperature. Colourless crystals

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were obtained, and further crystals were filtered off, washed with mother liquid, and dried under ambient conditions. Yield 48%.

Experimental details

Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program and refined with the ShelXL [3] refinement package. All hydrogen atoms were placed in calculated positions and refined isotropically with a riding model except for those bound to water molecules, which were initially located in a difference Fourier map and included in the final refinement and $U_{\text{iso}}(\text{H})$ equivalent to 1.5 times of $U_{\text{eq}}(\text{O})$.

Comment

Over the past decade, the research of coordination polymers (CPs) have attracted great attention because of their structural architectures [4–6], as well as their potential applications in the fields of catalysis [7], ion exchange [8], gas storage [9], fluorescence [10], magnetism [11]. In particular, lanthanide-based CPs are promising materials because of the intrinsic physical properties of the trivalent lanthanide ions, such as color-pure luminescence [12], large paramagnetism [13], and the fact that the electrostatic nature of their coordination chemistry allows a large variety of symmetries and structural patterns to be obtained. Most of the reported lanthanide-based CPs focus on the optimization of the choice of the ligand. The 2,6-dichloropyridine-4-carboxylic acid (Hdpcp) ligand that contains both pyridyl and carboxylate groups, is expected to form a structural diversity of coordination polymers.

The asymmetric unit of complex contains half a Tb(III) cation, one and half *dcpc* anion and one ligated water molecule. Each Tb(III) ion is coordinated by eight oxygen atoms: six oxygen atoms (O1, O1A, O2B, O2C, O3, O3A) from five different *dcpc* ligands and two oxygen atoms from two ligated water molecules (O1W, O1W(A)). The Tb–O bond distances fall in range of 2.290(29)–2.4889(19) Å, which are comparable to reported compounds. Two equivalent Tb(III) ions are doubly bridged by two equivalent carboxylate groups in the syn-anti bidentate bridging mode to give a 1D double chain motif along the *a* direction with the Tb–Tb distance of 4.9573(1) Å. Furthermore, one *dcpc* ligand coordinates Tb(III) ions *via* a chelating mode and decorate the chain from one side as a terminal ligand. The pyridyl groups, water molecules and chlorine atoms, which lie in the specific position, provide two type hydrogen bonding sites for interchain interactions. The paralleled chains are connected by H-bonds between coordination H₂O and pyridyl N atom of *dcpc* molecule [O(1W)–H(1WA)···N(2); d(O1···N2) = 3.122(4) Å; Angle (H–O···N) = 168.7°] to develop the 2D layer.

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