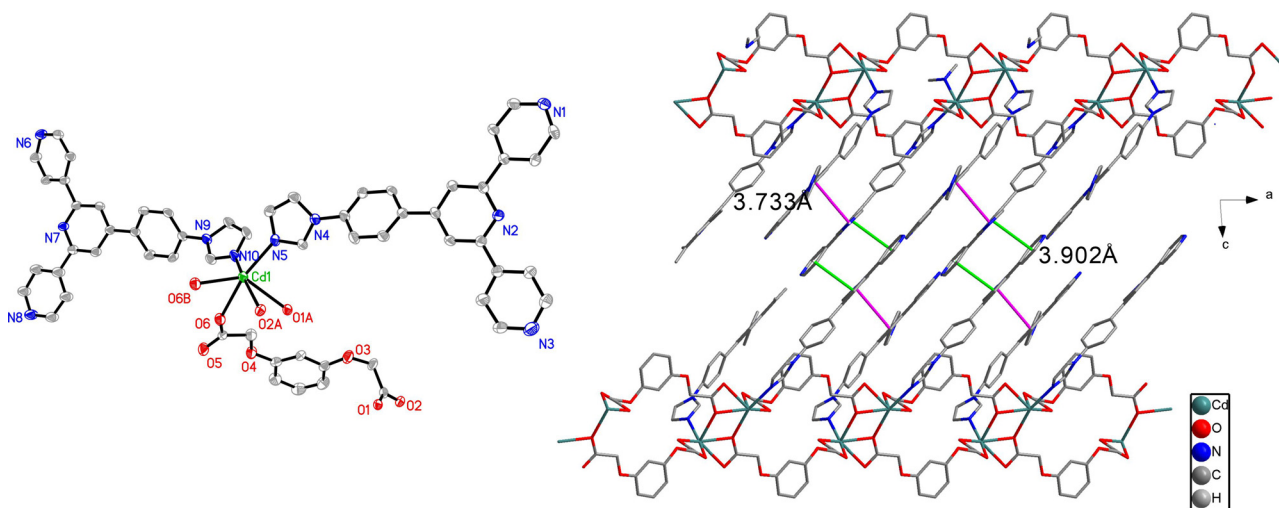


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The crystal structure of poly[(μ_3 -1,3-phenylenedioxydiacetate- $\kappa^5 O, O, O', O'', O'''$)-bis(4'-(4-(1*H*-imidazol-1-yl)phenyl)-4,2':6',4''-terpyridine-*kN*) cadmium(II)], $C_{58}H_{42}CdN_{10}O_6$



<https://doi.org/10.1515/ncrs-2023-0280>

Received July 13, 2023; accepted August 17, 2023;

published online September 5, 2023

Abstract

$C_{58}H_{42}CdN_{10}O_6$, triclinic, $P\bar{1}$ (no. 2), $a = 9.689(3) \text{ \AA}$, $b = 10.930(3) \text{ \AA}$, $c = 23.321(6) \text{ \AA}$, $\alpha = 80.838(5)^\circ$, $\beta = 84.261(5)^\circ$, $\gamma = 79.189(5)^\circ$, $V = 2388.7(12) \text{ \AA}^3$, $Z = 2$, $R_{gt}(F) = 0.0592$, $wR_{ref}(F^2) = 0.1561$, $T = 293 \text{ K}$.

CCDC no.: 2268377

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	$0.26 \times 0.21 \times 0.17 \text{ mm}$
Wavelength:	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
μ :	0.52 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0° , 99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12,104, 8356, 0.048
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5271
$N(\text{param})_{\text{refined}}$:	682
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

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

1 Source of material

The reagents were purchased from commercial sources and used without further purification. A mixture of $Cd(NO_3)_2 \cdot 4H_2O$ (0.031 g, 0.10 mmol), H_2pda (0.023 g, 0.10 mmol), and

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

Atom	x	y	z	U_{iso}^*/U_{eq}
C1	−0.2985 (7)	−0.5725 (6)	1.0172 (3)	0.0637 (19)
H1	−0.311105	−0.654303	1.015914	0.08 (2)*
C2	−0.2011 (7)	−0.5251 (6)	0.9772 (3)	0.0555 (17)
H2	−0.149788	−0.574414	0.950691	0.08 (2)*
C3	−0.1800 (6)	−0.4035 (5)	0.9768 (2)	0.0464 (15)
C4	−0.2573 (8)	−0.3403 (6)	1.0191 (3)	0.075 (2)
H4	−0.246258	−0.258914	1.022133	0.064 (19)*
C5	−0.3517 (8)	−0.3991 (7)	1.0573 (3)	0.079 (2)
H5	−0.402473	−0.353929	1.085465	0.08 (2)*
C6	−0.0788 (6)	−0.3442 (5)	0.9336 (2)	0.0442 (14)
C7	0.0078 (6)	−0.4109 (6)	0.8947 (2)	0.0448 (14)
H7	0.005102	−0.495508	0.894779	0.040 (15)*
C8	0.0989 (6)	−0.3510 (5)	0.8556 (2)	0.0426 (14)
C9	0.0949 (6)	−0.2251 (5)	0.8573 (2)	0.0478 (15)
H9	0.152962	−0.181075	0.830954	0.036 (14)*
C10	0.0064 (6)	−0.1635 (5)	0.8974 (2)	0.0445 (14)
C11	0.0015 (6)	−0.0282 (5)	0.9007 (2)	0.0485 (15)
C12	0.0910 (9)	0.0419 (7)	0.8676 (3)	0.088 (3)
H12	0.159693	0.004818	0.841822	0.11 (3)*
C13	0.0798 (9)	0.1680 (7)	0.8723 (3)	0.085 (3)
H13	0.141992	0.212994	0.848766	0.10 (3)*
C14	−0.0981 (8)	0.1611 (7)	0.9392 (3)	0.077 (2)
H14	−0.165580	0.200614	0.964680	0.08 (2)*
C15	−0.0938 (7)	0.0345 (6)	0.9371 (3)	0.067 (2)
H15	−0.157483	−0.008160	0.961001	0.09 (3)*
C16	0.1979 (6)	−0.4161 (5)	0.8123 (2)	0.0404 (13)
C17	0.1663 (6)	−0.5159 (5)	0.7886 (2)	0.0458 (15)
H17	0.083528	−0.546631	0.801223	0.07 (2)*
C18	0.2577 (6)	−0.5694 (5)	0.7462 (2)	0.0467 (15)
H18	0.236014	−0.635520	0.730122	0.08 (2)*
C19	0.3794 (6)	−0.5249 (5)	0.7282 (2)	0.0421 (14)
C20	0.4120 (6)	−0.4285 (6)	0.7519 (3)	0.0547 (17)
H20	0.495666	−0.399059	0.739647	0.058 (18)*
C21	0.3214 (6)	−0.3747 (6)	0.7938 (3)	0.0532 (16)
H21	0.344626	−0.309156	0.809737	0.038 (14)*
C22	0.5284 (7)	−0.7003 (6)	0.6814 (3)	0.0537 (16)
H22	0.498677	−0.768713	0.705152	0.10 (3)*
C23	0.6293 (6)	−0.7044 (6)	0.6372 (3)	0.0527 (16)
H23	0.681352	−0.777358	0.624996	0.050 (16)*
C24	0.5513 (6)	−0.5100 (6)	0.6426 (2)	0.0460 (15)
H24	0.538183	−0.422583	0.635380	0.043 (16)*
C25	1.3047 (7)	−1.5361 (6)	0.1678 (3)	0.0556 (17)
H25	1.347659	−1.571020	0.135213	0.050 (16)*
C26	1.2969 (6)	−1.4106 (5)	0.1670 (3)	0.0498 (15)
H26	1.332493	−1.362379	0.134326	0.07 (2)*
C27	1.2362 (6)	−1.3541 (5)	0.2146 (2)	0.0426 (14)
C28	1.1854 (6)	−1.4320 (5)	0.2615 (3)	0.0491 (15)
H28	1.143584	−1.399729	0.294839	0.030 (13)*
C29	1.1979 (6)	−1.5578 (6)	0.2579 (3)	0.0493 (15)
H29	1.163714	−1.608470	0.289983	0.064 (19)*
C30	1.2300 (6)	−1.2168 (5)	0.2157 (2)	0.0437 (14)
C31	1.1363 (6)	−1.1523 (5)	0.2538 (2)	0.0413 (13)
H31	1.072145	−1.193408	0.278244	0.046 (16)*
C32	1.1378 (6)	−1.0259 (5)	0.2556 (2)	0.0424 (14)
C33	1.2329 (6)	−0.9707 (5)	0.2161 (2)	0.0464 (15)

Table 2: (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H33	1.237499	−0.886247	0.215475	0.047 (16)*
C34	1.3214 (6)	−1.0395 (5)	0.1775 (2)	0.0444 (14)
C35	1.4210 (8)	−0.9830 (7)	0.1354 (3)	0.0830 (10)
C36	1.4285 (8)	−0.8590 (6)	0.1279 (3)	0.0830 (10)
H36	1.368560	−0.806084	0.150694	0.09 (2)*
C37	1.5242 (8)	−0.8098 (7)	0.0867 (3)	0.0830 (10)
H37	1.528094	−0.724736	0.084231	0.10 (2)*
C38	1.5970 (8)	−0.9918 (7)	0.0574 (3)	0.0830 (10)
H38	1.652803	−1.040674	0.031684	0.35 (9)*
C39	1.5081 (8)	−1.0514 (7)	0.0990 (3)	0.0830 (10)
H39	1.508947	−1.137370	0.101550	0.26 (6)*
C40	1.0429 (6)	−0.9537 (5)	0.2970 (2)	0.0412 (14)
C41	1.0090 (6)	−0.8249 (5)	0.2858 (3)	0.0487 (15)
H41	1.044238	−0.782549	0.251230	0.07 (2)*
C42	0.9240 (6)	−0.7579 (5)	0.3247 (2)	0.0461 (15)
H42	0.901909	−0.670656	0.316407	0.041 (15)*
C43	0.8716 (5)	−0.8184 (5)	0.3755 (2)	0.0387 (13)
C44	0.9032 (6)	−0.9451 (6)	0.3877 (3)	0.0483 (15)
H44	0.866239	−0.986274	0.422141	0.050 (16)*
C45	0.9906 (6)	−1.0142 (6)	0.3488 (3)	0.0495 (15)
H45	1.013842	−1.101273	0.357792	0.057 (18)*
C46	0.6637 (6)	−0.7595 (6)	0.4460 (3)	0.0548 (17)
H46	0.605963	−0.814256	0.439536	0.051 (16)*
C47	0.6363 (6)	−0.6779 (6)	0.4843 (3)	0.0547 (17)
H47	0.555539	−0.666563	0.509337	0.07 (2)*
C48	0.8358 (6)	−0.6581 (5)	0.4404 (2)	0.0496 (15)
H48	0.920751	−0.630485	0.428638	0.052 (17)*
C49	0.2411 (6)	0.2748 (5)	0.4287 (2)	0.0410 (13)
C50	0.2236 (6)	0.1414 (5)	0.4234 (3)	0.0568 (17)
H50A	0.148612	0.118108	0.451393	0.13 (3)*
H50B	0.194546	0.140838	0.384835	0.08 (2)*
C51	0.4436 (6)	0.0360 (6)	0.3860 (3)	0.0483 (15)
C52	0.5527 (6)	−0.0658 (5)	0.3949 (3)	0.0427 (14)
H52	0.557091	−0.116206	0.430990	0.07 (2)*
C53	0.6535 (6)	−0.0931 (5)	0.3515 (3)	0.0460 (14)
C54	0.6469 (7)	−0.0172 (6)	0.2976 (3)	0.0621 (18)
H54	0.714890	−0.034162	0.267464	0.061 (19)*
C55	0.5377 (8)	0.0838 (7)	0.2895 (3)	0.071 (2)
H55	0.532301	0.133981	0.253345	0.10 (3)*
C56	0.4371 (8)	0.1120 (7)	0.3333 (3)	0.0648 (19)
H56	0.365631	0.181512	0.327306	0.12 (3)*
C57	0.7761 (6)	−0.2680 (6)	0.4103 (3)	0.0556 (17)
H57A	0.714260	−0.329581	0.414249	0.047 (17)*
H57B	0.747836	−0.216362	0.441062	0.08 (2)*
C58	0.9257 (6)	−0.3346 (5)	0.4163 (3)	0.0427 (14)
Cd1	0.81726 (4)	−0.51625 (4)	0.54812 (2)	0.04025 (16)
N1	−0.3753 (6)	−0.5126 (5)	1.0570 (2)	0.0627 (15)
N2	−0.0817 (5)	−0.2204 (4)	0.93546 (19)	0.0469 (12)
N3	−0.0121 (7)	0.2281 (5)	0.9073 (3)	0.0694 (16)
N4	0.4783 (5)	−0.5755 (4)	0.68448 (19)	0.0446 (12)
N5	0.6433 (5)	−0.5843 (4)	0.61321 (19)	0.0445 (12)
N6	1.2548 (5)	−1.6129 (4)	0.2122 (2)	0.0549 (13)
N7	1.3203 (5)	−1.1630 (4)	0.17835 (19)	0.0427 (11)
N8	1.6082 (7)	−0.8738 (5)	0.0515 (3)	0.0830 (10)
N9	0.7914 (5)	−0.7476 (4)	0.41814 (19)	0.0421 (11)
N10	0.7458 (5)	−0.6138 (5)	0.4808 (2)	0.0494 (13)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
O1	0.3332 (4)	0.2938 (3)	0.45665 (15)	0.04025 (16)
O2	0.1483 (4)	0.3587 (3)	0.40540 (16)	0.0452 (9)
O3	0.3471 (4)	0.0486 (4)	0.4326 (2)	0.0655 (12)
O4	0.7630 (4)	−0.1911 (4)	0.35530 (17)	0.0556 (11)
O5	1.0198 (5)	−0.3222 (4)	0.37793 (19)	0.0686 (13)
O6	0.9457 (4)	−0.4045 (4)	0.46343 (17)	0.0497 (10)

4264-imphtpy (0.038 g, 0.10 mmol) was dispersed in mixed DMF (4 mL) and CH_3CN (4 mL) solutions in a 16 mL Teflon-lined stainless steel autoclave, which was heated for 3 d at 393 K under autogenous pressure and slowly cooled to room temperature. Pale yellow block crystals were obtained.

2 Experimental details

The structure was solved with the SHELXT-2018 program. All H-atoms from C atoms were positioned with idealized geometry and refined isotropically ($U_{iso}(H) = 1.2U_{eq}(C)$) using a riding model with C–H = 0.93 and 0.97 Å.

3 Comment

In the past decades, a mixed strategy with N-heterocycle rings N-donor ligand and polycarboxylate O-donor ligand has been widely accepted for the diverse structures and wide applications of metal-organic frameworks (MOFs) [5–7]. As a rigid planar ligand, the tridentate ligand 4'-(4-(imidazol-1-yl)phenyl)-4,2':6',4"-terpyridine (imphtpy) [8, 9] has been adopted for possessing both pyridine and imidazole functional groups, which is rarely used in the realm of MOFs relative to 4'-(4-pyridyl)-4,2':6',4"-terpyridine (pytpy) [10, 11]. A Cd(II) coordination complex was obtained successfully along with 1,3-phenylenedioxidiacetate(pda^{2-}) [12–14] and its structure has been determined.

The ORTEP diagram is presented in the left part of the figure. The asymmetric unit contains one Cd(II) ion, two imphtpy ligands, and one completely deprotonated pda^{2-} anion. As shown in ORTEP Figure, Cd1 is seven coordinated by two imidazole nitrogen atoms (Cd1–N5, 2.302(4) Å, Cd1–N5, 2.261(5) Å) from two individual imphtpy ligands and five oxygen atoms which are derived from one chelating carboxylate (O1#1 and O2#1, #1, symmetry code: #1, 1 – *x*, –*y*, 1 – *z*), and two monodentate bridging carboxylate (O6 and O6#2, symmetry code: #2, 2 – *x*, –1 – *y*, 1 – *z*) of three different pda^{2-} anions (Cd1–O, 2.283(3)–2.595(3) Å) to furnish a

distorted octahedral geometry. Cd(II) to O/N distances and bond angles are within the normal range [15] except a distance of 2.667 Å between Cd1 and O5 indicating the existence of a weak interaction between them.

Every two neighbouring Cd(II) ions are linked by O6 atoms to form dimers, which are further connected by residue of two pda^{2-} anions to generate one dimensional (1D) chain with imphtpy as a monodentate ligand through its terminal imidazole nitrogen atom. As there are no classic hydrogen bonds in this complex, the 3D structure is obtained through π – π interactions with Cg–Cg (the aromatic rings center was defined as Cg) distances of 3.902 Å and 3.733 Å between pyridine rings among adjacent chains demonstrated in the right part of the figure along with van der Waals forces.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by the 2022 Innovation and Entrepreneurship Training Program for Hengyang Normal University students (No. cxcy2022033), and the Innovation Platform Open Fund Project of Hunan Provincial Education Department of China (No. 20K016).

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

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