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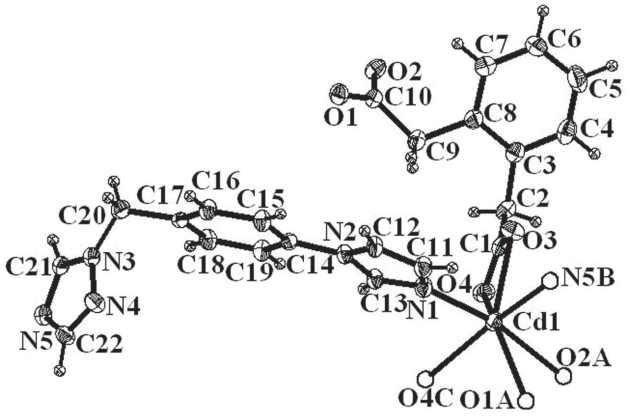
Crystal structure *poly*-(μ$_2$-1-(4-(1*H*-imidazol-1-yl)benzyl)-1*H*-1,2,4-triazolyl-κ^2*NN*^{1}:*N*^{2}*N*)-(μ$_3$-2,2′-(1,2-phenylene)diacetato-κ^5-*O*^{1},*O*^{2}:*O*^{2}:*O*^{3},*O*^{4}) cadmium(II), $C_{22}H_{19}CdN_5O_4$



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Abstract

$C_{22}H_{19}CdN_5O_4$, monoclinic, $P2_1/c$, $a = 11.7546(16)$ Å, $b = 19.769(3)$ Å, $c = 9.5012(13)$ Å, $\beta = 106.252(2)°$, $V = 2119.6(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0348$, $wR_{ref}(F^2) = 0.0814$, $T = 296(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The mixture of 1,2-phenylenediacetic acid (phda) (0.1 mmol, 19.4 mg), 1-(imidazo-1-ly)-4-(1,2,4-triazol-1-yl-methyl)benzene

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Table 1: Data collection and handling.

Crystal:	Colourless blocks
	Size 0.38 × 0.29 × 0.18 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	10.7 cm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω
$2\theta_{max}$, completeness:	51°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9295, 3906, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3129
$N(param)_{refined}$:	289
Programs:	SHELX [9], Bruker programs [10]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.59076(2)	0.074350(13)	0.60435(3)	0.03729(11)
N1	0.4970(3)	0.15505(15)	0.4438(3)	0.0410(7)
N2	0.3842(2)	0.20453(13)	0.2458(3)	0.0328(6)
N3	−0.0978(3)	0.21857(16)	−0.2710(3)	0.0396(7)
N4	−0.1442(3)	0.23805(18)	−0.1618(4)	0.0524(9)
N5	−0.2562(3)	0.15714(16)	−0.2996(3)	0.0429(8)
O1	0.5596(2)	0.09669(14)	−0.1553(3)	0.0504(7)
O2	0.6595(3)	0.00746(13)	−0.1867(3)	0.0507(7)
O3	0.7310(3)	0.05665(15)	0.4532(3)	0.0617(8)
O4	0.6054(3)	−0.02140(16)	0.4610(3)	0.0622(8)
C1	0.6879(3)	−0.0004(2)	0.4185(3)	0.0385(9)
C2	0.7434(4)	−0.04559(19)	0.3275(4)	0.0456(10)
H2A	0.7853	−0.0817	0.3899	0.055*
H2B	0.6802	−0.0662	0.2513	0.055*
C3	0.8278(3)	−0.01249(18)	0.2562(4)	0.0385(8)
C4	0.9489(4)	−0.0195(2)	0.3189(4)	0.0528(11)
H4	0.9762	−0.0457	0.4028	0.063*
C5	1.0298(4)	0.0118(3)	0.2590(5)	0.0657(13)
H5	1.1107	0.0064	0.3021	0.079*
C6	0.9901(4)	0.0510(3)	0.1354(5)	0.0683(13)
H6	1.0436	0.0732	0.095	0.082*
C7	0.8707(4)	0.0569(3)	0.0716(5)	0.0636(13)
H7	0.8442	0.0827	−0.0131	0.076*
C8	0.7875(3)	0.0254(2)	0.1296(4)	0.0472(10)
C9	0.6551(4)	0.0329(3)	0.0567(4)	0.0709(15)
H9A	0.6157	−0.0083	0.0730	0.085*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H9B	0.6251	0.0696	0.1039	0.085*
C10	0.6223(3)	0.0467(2)	−0.1064(4)	0.0409(9)
C11	0.5193(3)	0.22341(19)	0.4513(4)	0.0438(9)
H11	0.5734	0.2452	0.5284	0.053*
C12	0.4514(3)	0.25416(18)	0.3307(4)	0.0419(9)
H12	0.4503	0.3001	0.3091	0.050*
C13	0.4146(3)	0.14530(18)	0.3197(4)	0.0394(9)
H13	0.3815	0.1035	0.2867	0.047*
C14	0.2934(3)	0.21390(16)	0.1108(3)	0.0303(7)
C15	0.2670(3)	0.27860(18)	0.0570(4)	0.0390(9)
H15	0.3094	0.3152	0.1067	0.047*
C16	0.1770(3)	0.28888(18)	−0.0717(4)	0.0405(9)
H16	0.1597	0.3325	−0.1078	0.049*
C17	0.1133(3)	0.23544(18)	−0.1461(3)	0.0348(8)
C18	0.1426(3)	0.17085(19)	−0.0927(4)	0.0442(9)
H18	0.1012	0.1341	−0.1434	0.053*
C19	0.2319(3)	0.15979(18)	0.0343(4)	0.0416(9)
H19	0.2508	0.1159	0.0684	0.050*
C20	0.0126(3)	0.2478(2)	−0.2845(4)	0.0466(10)
H20A	0.0024	0.2960	−0.3017	0.056*
H20B	0.0324	0.2277	−0.3678	0.056*
C21	−0.1659(3)	0.17082(19)	−0.3511(4)	0.0408(9)
H21	−0.1516	0.1500	−0.4323	0.049*
C22	−0.2382(3)	0.2002(2)	−0.1847(4)	0.0494(10)
H22	−0.2896	0.2026	−0.1261	0.059*

(itmb) (0.2 mmol, 45.0 mg), $Cd(OAc)_2{\cdot}2H_2O$ (0.1 mmol, 26.7 mg), NaOH (0.1 mmol), and H_2O (6.0 mL) were placed in a 23 mL Teflon liner stainless steel reactor. The vessel was heated to 393 K for 4 days, and then slowly cooled to room temperature. Colorless crystals were obtained, filtered off, washed with mother liquid, and dried under ambient conditions (yield 58%).

Experimental details

Carbon-bound hydrogen atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C).

Discussion

Assemble of coordination polymers (CPs) based on metal and organic building blocks has been expanding in recent years, not only because of fascinating structural diversities and framework topologies, but also due to their potential applications in the fields of gas storage, catalysis, magnetism, luminescence, and so on [1–4]. The extension of framework topologies or chemical compositions for such materials has been tremendously accelerated because new framework-forming elements can adopt various coordination geometries to produce some previously unknown framework structures. Among them, the CPs which were built by flexible ligands can afford a good opportunity to enrich the structural and functional diversities of CPs. Our previous work has demonstrated that the flexible aromatic-carboxylate ligands can freely rotate to meet the requirement of coordination geometries of metal ions [5, 6], thus improving the framework flexibility and associated properties for CPs. Furthermore, the dipyridyl-type molecules are widely used coligands that can pillar polycarboxylate motifs into high dimensional structures [7], while there is relatively little effort to synthesize CPs derived from polydentate aromatic nitrogen heterocyclic coligands, such as pyrazoles, imidazoles, and triazoles. In fact, the introduction of such nitrogen-rich coligands in carboxylate systems is beneficial for the formation of new CPs based on the following considerations: (i) they can act as multi-bridges between metal centers, thus supplying for more ligated modes. (ii) The prototropy and conjugation between aromatic nitrogen heterocyclic alter the electron density in different parts of the molecules, and make the ligands more flexible [8].

The asymmetric unit of the title structure contains one Cd atom, one phda dianion and one itmb coligand. The Cd atom displays a highly distorted pentagonal bipyramid with five oxygen atoms from carboxylate group belonging to three phda ligands [Cd—O: 2.3493(18) Å—2.5135(16) Å] and two nitrogen atoms from two itmb ligands [Cd—N: 2.346(3) Å and 2.369(3) Å]. Each phda anion acts as a pentadentate ligand connecting three cadmium ions: one carboxylic group tri-dentately bridges two cadmium atoms to accelerate sharing-edge cadmium dimers with the Cd···Cd distance of 3.8416(5) Å, and the other bidentatly chelating coordination mode generate 1D ribbon-like chains. Such 1D $[(Cd_2)(phda)]_n$ arrays are further extended by itmb spacers to afford a 2D polymeric bilayer. Interestingly, no H-bonds are observed between the layers, which stack in a slightly off-set parallel fashion and are cohered only by van der Waals force.

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