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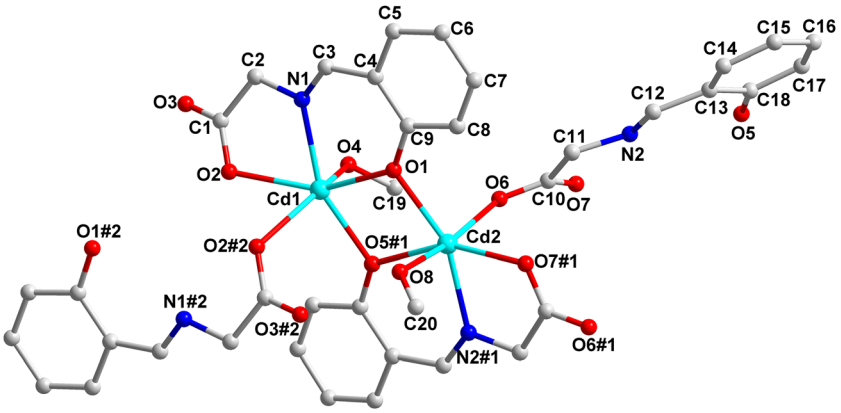
Crystal structure of poly[dimethanol-κ^{1}O-(μ$_2$-(*E*)-2-((2-oxidobenzylidene)amino)acetato)-(μ$_3$-(*E*)-2-((2-oxidobenzylidene)amino)acetato) dicadmium(II)], $C_{20}H_{22}Cd_2N_2O_8$



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Abstract

$C_{20}H_{22}Cd_2N_2O_8$, monoclinic, $P2_1/c$ (no. 14), a = 13.131(3) Å, b = 8.2018(16) Å, c = 20.973(4) Å, β = 103.59(3)°, V = 2195.5(8) Å^3, Z = 4, $R_{gt}(F)$ = 0.0260, w$R_{ref}(F^2)$ = 0.0609, T = 293(2) K.

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A part of the title structure is shown in the figure. 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:	Yellow prism
Size:	0.23 × 0.21 × 0.21 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.99 mm^{-1}
Diffractometer, scan mode:	Rigaku Saturn, ω
θ_{max}, completeness:	26.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22832, 4300, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 4115
$N(param)_{refined}$:	296
Programs:	SHELX [1, 2], CrystalClear [3]

Source of material

All reagents used in this paper were purchased from commercial sources and used without further purification. A methanol solution (3 mL) of {[1-(2-hydroxyphenyl) methylidene]amino}acetic acid (H_2L, 0.05 mmol) was added dropwise to an aqueous solution (3 mL) of $CdCl_2$·2.5H_2O (0.05 mmol). The resulting solution was placed in a 25 mL Teflon-lined stainless steel reactor, which was sealed and heated to 353 K for 72 h. After the mixture was cooled to room temperature at a rate of 5 K h^{-1}, yellow crystals of the title compound were obtained (yield 26%, based on Cd).

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.38743 (2)	0.59031 (2)	0.43339 (2)	0.02857 (7)
Cd2	0.16756 (2)	0.43739 (3)	0.32271 (2)	0.03002 (7)
N1	0.49929 (18)	0.7484 (3)	0.39478 (12)	0.0330 (5)
N2	−0.04881 (19)	0.7942 (3)	0.13776 (12)	0.0396 (6)
O1	0.31999 (15)	0.5571 (3)	0.32522 (10)	0.0363 (5)
O2	0.52666 (15)	0.6562 (2)	0.52312 (10)	0.0340 (5)
O3	0.62212 (17)	0.8746 (3)	0.55960 (10)	0.0460 (6)
O4	0.30615 (18)	0.8295 (3)	0.46241 (13)	0.0490 (6)
H4	0.3300 (17)	0.9249 (14)	0.4559 (19)	0.073*
O5	−0.23809 (15)	0.9677 (2)	0.06770 (9)	0.0312 (4)
O6	0.08170 (19)	0.6789 (3)	0.30385 (11)	0.0543 (7)
O7	−0.05741 (17)	0.8317 (3)	0.26685 (10)	0.0433 (5)
O8	0.2723 (2)	0.2003 (3)	0.31689 (12)	0.0571 (7)
H8	0.323 (2)	0.185 (3)	0.3503 (12)	0.086*
C1	0.5737 (2)	0.7865 (3)	0.51453 (15)	0.0332 (6)
C2	0.5721 (2)	0.8410 (4)	0.44522 (15)	0.0372 (7)
H2A	0.553056	0.955407	0.440740	0.045*
H2B	0.642100	0.830372	0.437985	0.045*
C3	0.5086 (2)	0.7631 (4)	0.33604 (15)	0.0370 (7)
H3	0.565983	0.823353	0.330660	0.044*
C4	0.4421 (2)	0.6990 (4)	0.27669 (14)	0.0338 (6)
C5	0.4701 (3)	0.7433 (4)	0.21797 (16)	0.0453 (8)
H5	0.531445	0.802034	0.220449	0.054*
C6	0.4097 (3)	0.7024 (4)	0.15751 (16)	0.0503 (9)
H6	0.429079	0.734437	0.119491	0.060*
C7	0.3196 (3)	0.6130 (4)	0.15405 (15)	0.0433 (8)
H7	0.277405	0.586051	0.113261	0.052*
C8	0.2913 (2)	0.5630 (4)	0.21016 (14)	0.0359 (7)
H8A	0.231257	0.500033	0.206444	0.043*
C9	0.3506 (2)	0.6048 (3)	0.27284 (14)	0.0302 (6)
C10	0.0164 (2)	0.7458 (4)	0.25817 (15)	0.0384 (7)
C11	0.0324 (3)	0.7200 (5)	0.18993 (17)	0.0530 (9)
H11A	0.099886	0.764928	0.187881	0.064*
H11B	0.034393	0.603764	0.181760	0.064*
C12	−0.0426 (3)	0.7739 (5)	0.07951 (16)	0.0487 (9)
H12	0.013751	0.711370	0.073912	0.058*
C13	−0.1117 (2)	0.8352 (4)	0.01933 (15)	0.0431 (8)
C14	−0.0835 (3)	0.7953 (6)	−0.03923 (19)	0.0695 (13)
H14	−0.024489	0.730957	−0.036936	0.083*
C15	−0.1388 (4)	0.8465 (6)	−0.09967 (18)	0.0750 (14)
H15	−0.117695	0.818192	−0.137561	0.090*
C16	−0.2267 (3)	0.9413 (5)	−0.10260 (17)	0.0573 (10)
H16	−0.265132	0.978521	−0.143013	0.069*
C17	−0.2580 (3)	0.9813 (4)	−0.04664 (15)	0.0426 (7)
H17	−0.317649	1.045049	−0.050170	0.051*
C18	−0.2032 (2)	0.9295 (3)	0.01570 (14)	0.0301 (6)
C19	0.1956 (3)	0.8534 (5)	0.4556 (2)	0.0612 (10)
H19A	0.158636	0.823275	0.412040	0.092*
H19B	0.182318	0.966020	0.463099	0.092*
H19C	0.172148	0.786904	0.486948	0.092*
C20	0.2450 (4)	0.0473 (5)	0.2900 (2)	0.0841 (16)
H20A	0.306548	−0.019632	0.296140	0.126*
H20B	0.195682	−0.002453	0.311291	0.126*
H20C	0.213940	0.058096	0.243955	0.126*

Experimental details

Hydrogen atoms bound to C atoms were positioned geometrically and refined as riding atoms, with C–H = 0.93 Å (CH, aromatic) or 0.96 Å (CH_3) or 0.97 Å (CH_2). Hydrogen atoms on CH_3OH were found according to the residual electron-density peaks with O–H = 0.866(9) Å (O4–H4) or 0.858(9) Å(O8–H8). H atoms were refined with U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(C, O) for methyl H atoms and OH.

Comment

Schiff-bases are considered as good ligands and have been widely used in the synthesis of new coordination compounds (CPs) [4, 5]. Among the reported literatures, amino acid Schiff bases and their corresponding CPs have received much attention for their medicinal and pharmaceutical applications, especially for their antibacterial and anticancer activity [6–8]. The above results stimulated our research interest on the N-salicylideneglycinato-metal CPs. Up to now, a series of N-salicylideneglycinato-metal CPs have been synthesized and characterized, such as Cr(III), Ni(II), Cu(II) and zinc(II) CPs [8–10]. In order to further enrich the categories and numbers of CPs based on this ligand, we continue to use {[1-(2-hydroxyphenyl) methylidene]amino}acetic acid (H_2L) as ligand to react with $CdCl_2$·2.5H_2O and obtained the new CP [$Cd_2L_2(CH_3OH)_2$].

The asymmetric unit of the title structure consists of two Cd(II) cations, two 2-((2-oxidobenzylidene)amino)acetate anions (L^{2-}) and two methanol molecules. Each Cd1 cation is six-coordinated and located in a distorted octahedral $CdNO_5$ coordination environment formed by one N atom (N1) and four O atoms (O1, O2, O2#2 and O5#1) from two L^{2-} anions and by one methanol molecule (O4). Each Cd2 cation is also six-coordinated by N2#1, O1, O5#1, O6 and O7#1 from two L^{2-} anions together with one methanol molecule (O8), exhibiting a deformed $CdNO_5$ octahedral geometry. The bond lengths of Cd–O are in the range of 2.1990(19)–2.402(2) Å and the bond lengths of Cd–N are 2.248(2) and 2.260(3) Å, respectively. It is known from the literature that these bond lengths are similar to those reported in other Cd(II) complexes [11–13]. There are two kinds of crystallographically independent L^{2-} anion ligands. One L^{2-} anion ligand coordinates to Cd(II) cations in tridentate mode, while the other L^{2-} anion ligand coordinates to Cd(II) cations in tetradentate mode. Cd1 and Cd2 cations are furthermore connected by L^{2-} anion ligands (see the figure) to form a two-dimensional layered structure with Cd1–Cd1 distance of 3.8547(12) Å, Cd2–Cd2 distance of 6.2591(13) Å and Cd1–Cd2

distance of 3.4854(11) Å. In addition, there are O–H···O hydrogen bonds between hydroxyl and carboxylate groups. Adjacent layers are further packed through van der Waals forces, resulting in a three-dimensional architecture.

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

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