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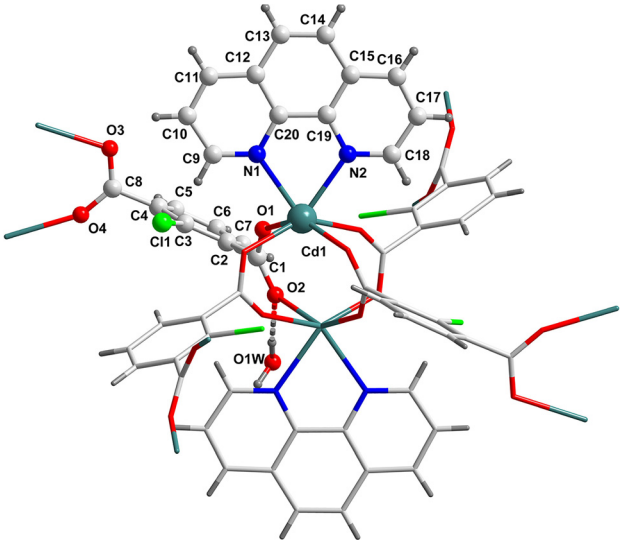
The crystal structure of poly [(1,10-phenanthroline-κ^2*N*,*N*′)-(μ_4-2-chlorobenzene-1,3-dicarboxylato-κ^5*O*:*O*′:*O*″:*O*‴) cadmium(II)] monohydrate, $C_{20}H_{13}CdClN_2O_5$



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Abstract

$C_{20}H_{13}CdClN_2O_5$, monoclinic, $P2_1/n$ (no. 14), a = 11.4994(9) Å, b = 13.1171(10) Å, c = 13.0233(10) Å, β = 109.126(8)°, V = 1856.0(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0341, $wR_{ref}(F^2)$ = 0.0779, T = 293 K.

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

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Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.25 × 0.16 × 0.11 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.36 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	26.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8934, 3648, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3019
$N(param)_{refined}$:	265
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3, 4]

Source of materials

A mixture of 0.0198 g $Cd(NO_3)_2 \cdot 4H_2O$ (0.10 mmol), 0.0198 g 1,10-phenanthroline monohydrate (0.10 mmol), 0.0200 g 2-chlorobenzene-1,3-dicarboxylic acid (0.10 mmol), 0.008 g NaOH (0.20 mmol) was added to 10 mL water and stirred to form a clear solution, then transferred to a 25 mL Teflon-lined autoclave and heated at 413 K in an oven for 72 h, cooled to room temperature. Colorless crystals were washed by deionized water and air-dried, yield 43% (based on 2-chlorobenzene-1,3-dicarboxylic acid).

Experimental details

The structure was solved by Direct Methods with the SHELXS-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 = 0.930 Å. The H-atoms from O atoms positioned with Q peaks refined isotropically with the distance of O—H = 0.850 Å (U_{iso}(H) = 1.5U_{eq}(O)). No suitable receptor was found for O1W—H1WB after careful refinement, resulting a B alert in the checkcif report.

Comment

Substituents play a key role in the synthesis of metal-organic complexes. Many Cd(II) complexes based on isophthalic acid

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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}
C1	0.6360 (3)	0.4471 (3)	0.7066 (3)	0.0310 (8)
C2	0.7252 (3)	0.4170 (3)	0.8154 (2)	0.0264 (7)
C3	0.7879 (3)	0.3245 (2)	0.8325 (3)	0.0271 (7)
C4	0.8766 (3)	0.3002 (3)	0.9311 (3)	0.0286 (8)
C5	0.9014 (3)	0.3704 (3)	1.0142 (3)	0.0444 (10)
H5	0.961244	0.356168	1.080536	0.053*
C6	0.8383 (4)	0.4618 (3)	1.0000 (3)	0.0501 (11)
H6	0.854872	0.508299	1.056891	0.060*
C7	0.7508 (4)	0.4841 (3)	0.9012 (3)	0.0397 (9)
H7	0.708302	0.545582	0.892443	0.048*
C8	0.9412 (3)	0.1985 (3)	0.9473 (3)	0.0350 (8)
C9	0.8172 (4)	0.2793 (3)	0.4820 (3)	0.0515 (11)
H9	0.762374	0.247995	0.510980	0.062*
C10	0.9216 (5)	0.2247 (4)	0.4801 (4)	0.0697 (14)
H10	0.936048	0.158934	0.508066	0.084*
C11	1.0008 (4)	0.2699 (5)	0.4365 (4)	0.0727 (16)
H11	1.069858	0.234617	0.433903	0.087*
C12	0.9793 (3)	0.3694 (4)	0.3956 (3)	0.0501 (11)
C13	1.0589 (4)	0.4216 (5)	0.3493 (4)	0.0720 (16)
H13	1.129391	0.389251	0.345710	0.086*
C14	1.0331 (4)	0.5172 (5)	0.3108 (4)	0.0737 (17)
H14	1.086825	0.549670	0.281352	0.088*
C15	0.9258 (4)	0.5702 (4)	0.3138 (3)	0.0536 (12)
C16	0.8968 (5)	0.6689 (4)	0.2771 (3)	0.0694 (15)
H16	0.946414	0.703985	0.245235	0.083*
C17	0.7956 (5)	0.7142 (4)	0.2878 (4)	0.0704 (14)
H17	0.776304	0.781093	0.264727	0.085*
C18	0.7206 (4)	0.6603 (3)	0.3336 (3)	0.0514 (11)
H18	0.650991	0.692135	0.339877	0.062*
C19	0.8469 (3)	0.5206 (3)	0.3599 (3)	0.0349 (9)
C20	0.8726 (3)	0.4183 (3)	0.4007 (3)	0.0363 (9)
Cd1	0.61898 (2)	0.46910 (2)	0.44675 (2)	0.02491 (10)
Cl1	0.75343 (9)	0.23209 (7)	0.73064 (7)	0.0468 (3)
N1	0.7938 (3)	0.3728 (2)	0.4446 (2)	0.0356 (7)
N2	0.7446 (3)	0.5658 (3)	0.3685 (2)	0.0376 (7)
O1	0.6733 (2)	0.4381 (2)	0.62756 (19)	0.0425 (6)
O2	0.5343 (2)	0.4825 (2)	0.70580 (19)	0.0419 (7)
O3	1.0328 (2)	0.1897 (2)	0.9177 (2)	0.0507 (7)
O4	0.8970 (3)	0.1315 (2)	0.9909 (2)	0.0545 (7)
O1W	0.4311 (7)	0.5239 (5)	0.8766 (5)	0.142 (2)
H1WA	0.461801	0.508497	0.827478	0.213*
H1WB	0.374252	0.480311	0.869334	0.213*

and its derivatives combining 1,10-phe-nanthroline have been published elsewhere, including isophthalate [5, 6], 5-substituted isophthalate (such as 5-methyl [7], 5-hydroxy [8], 5-iodo [9], 5-nitro [10], 5-ethoxyl [11], 5-phenyl [12], 5-(benzyloxy) [13], 5–ferrocene [14], 5-(((4-methylphenyl)sulfonyl)amino) [15], 5-((1H-benzimidazol-2-ylsulfanyl) [16] and 5-(1,3-benzo-thiazol-2-yl)) [17], 4-substituted isophthalic acid (4-(pyridin-4-yl)) [18], multi-substituted isophthalic acid (4,6-dimethyl-5-nitro [19] and 5-amino-2,4,6-triiodo [20]), and 2-substituted isophthalate (2-hydroxy [21]). Almost all the cadmium complexes mentioned above are 1D structures. It's interesting that when the target ligand 2-substituted isophthalic acid, 2-chlorobenzene-1,3-dicarboxylic acid was used to synthesize Cd(II) complexes, a novel 2D structure was obtained.

The title compound crystallizes in the monoclinic space group $P2_1/n$ (no. 14), with the formula $C_{20}H_{13}CdClN_2O_5$. The asymmetric unit is composed of one Cd(II) cation, one 1,10-phena-nthroline, one completely deprotonated 2-chlorobenzene-1,3-dicarboxylate, and one crystal water molecule. The Cd1 center is coordinated by two nitrogen atoms (N1, N2) form a bidentate 1,10-phenanthroline and four carboxyl oxygen atoms (O1, O2 (code: $1 - x$. $1 - y$, $1 - z$), O3 (code: $-0.5 + x$, $0.5 - y$, $-0.5 + z$), O4 (code: $1.5 - x$, $0.5 + y$, $1.5 - z$)) of four different 2-chlorobenzene-1,3-dicarboxylate anions in a trigonal prismatic geometry. All of the carboxyl groups are bismonodentate, linking two crystallographic Cd(II) to form a dimer, which is decorated by two 1,10-phenanthroline to generate the second building units (SBUs). The SBUs are bridged together by the μ_4-2-chlorobenzene-1,3-dicarboxy-late anions to generate a 2D structure. The distances of the Cd1—N, Cd—O, and the two Cd(II) cations in the same SBUs are comparable with the similar di–Cd(II) complex, respectively [22, 23].

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