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Crystal structure of aquabis(acrylato-*O,O'*)(1,10-phenanthroline-*N,N'*)-cadmium(II), $Cd(H_2O)(C_3H_3O_2)_2(C_{12}H_{10}N_2)$

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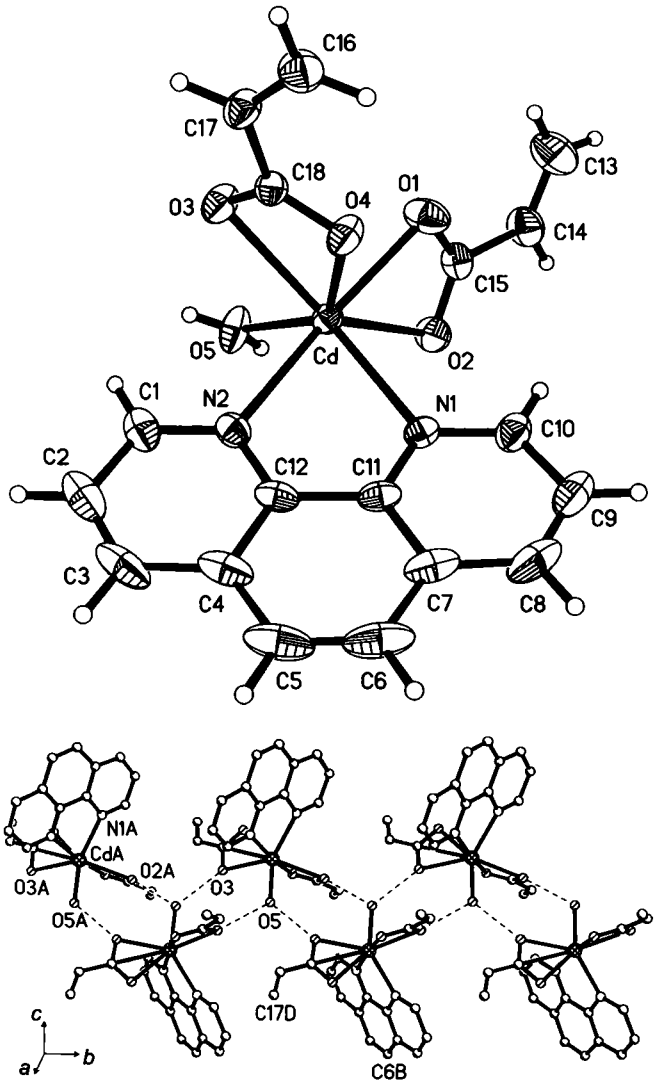

Abstract

$C_{18}H_{15}CdN_2O_5$, monoclinic, $P12_1/n1$ (no. 14), a = 9.9973(5) Å, b = 9.0433(4) Å, c = 19.4849(8) Å, β = 101.235(2)°, V = 1727.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.025, $wR_{ref}(F^2)$ = 0.065, T = 273 K.

Source of material

The title complex was prepared analogously to the synthesis of $[CdCl(C_3H_3O_2)(C_{12}H_{10}N_2)_2]$ except for the pH of this solution controlled in the range of 8-9 [1]. Freshly prepared precipitate $Cd(OH)_2$, which was obtained from 1.0 ml (1.0 M) NaOH to a stirred aqueous solution of $CdCl_2 \cdot 6H_2O$ (0.269 g, 1.0 mmol) in 5 ml H_2O, was added to a stirred aqueous solution of 1,10-phenanthroline monohydrate (0.198 g, 1.0 mmol) and acrylic acid (0.144 g, 2.0 mmol) in 20 ml H_2O/ethanol (1:1, *v/v*). The mixture was further stirred for ca. 30 min at room temperature, and the pH of the mixture solution was controlled in the range of 8-9 with 1 M NaOH solution. The mixture then was further stirred for another 2 h and filtered off and the resultant filtrate was allowed to stand by slow evaporation at room temperature. The colorless block-like crystals were obtained 30 days later (yield 80 % based on the $Cd(OH)_2$ input).

Discussion

The title structure consists of $Cd(H_2O)(C_3H_3O_2)_2(C_{12}H_{10}N_2)$ complex molecules, where the seven-coordinated Cd atom adopts a distorted monocapped octahedral environment formed by two nitrogen atoms of an 1,10-phenanthroline ligand and five oxygen atoms from one aqua ligand and two acrylato anion groups with the acrylato O2 atom residing at the cap site and the axial apical positions occupied by one aromatic pyridyl atoms (N2) and one acrylato O atom (O1) (figure, top). The equatorial plane sites are defined by one aromatic pyridyl N atom (N1), one aqua ligand (O5) and two acrylato O atoms (O3, O4) with the pyridyl N1 atom shifted from the basal plane of 0.9999(3) Å. The Cd—N2 length (2.3706(2) Å) is slightly longer than the Cd—N1 length (2.3676(2) Å), but shorter than those found in the compound of $[CdCl(C_3H_3O_2)(C_{12}H_{10}N_2)_2]$, where the Cd—N distances fall in the range of 2.3984(2) Å – 2.5619(2) Å [1]. The acrylato anion ligands adopt bidentate chelating mode. The Cd—O2 distance (2.5753(2) Å) is significantly longer than other Cd—O bond distances (d(Cd—O) = 2.298(2) Å – 2.4555(2) Å), indicating that Cd—O2 bonding interaction is weaker than other Cd—O bonding interactions. The mean Cd—O distance (2.4248(9) Å) is slightly longer than those in the previously reported bidentate chelate carboxylato compound [2].

Within the crystal structure, the title compound molecules are held together by weak intermolecular nonclassical C–H···O hydrogen-bonding interactions and π-π interactions. The coordinated water molecules functioning as hydrogen-bond donors contribute H atoms to the acrylato O atoms ($O2^i$, $O3^{ii}$) of neighboring complex molecules to form intermolecular hydrogen bonds (d(O5···$O2^i$) = 2.730 Å, ∠O5–H5b···$O2^i$ = 163.0°; d(O5···$O3^{ii}$) = 2.731 Å, ∠O5–H5a···$O3^{ii}$ = 152.0°) (symmetry codes: (i) ½–x,–½+y,½–z; (ii) ½–x,½+y,½–z). Along the [010] direction, the complex molecules are interlinked via the intermolecular hydrogen-bonding interactions into 1D double chains. The resulting chains are further held by π-π interactions between the 1,10-phenanthroline ligands of neighboring double chains into layers parallel to ($\bar{1}01$) with an average interplanar distance of 3.4522(5) Å (figure, bottom).

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Table 1. Data collection and handling.

Crystal:	colorless block, size 0.25 × 0.30 × 0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.96 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD II, ω
$2\theta_{max}$:	56.82°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16347, 4265
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3628
$N(param)_{refined}$:	235
Programs:	SHELXS-97 [3], SHELXL-97 [4]

Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{iso}
H(5A)	4e	0.8007	0.4087	0.7435	0.084
H(1A)	4e	0.9552	0.1039	0.8526	0.068
H(2A)	4e	1.1532	0.0241	0.9246	0.088
H(3A)	4e	1.2046	0.1051	1.0370	0.094
H(5B)	4e	1.1373	0.2658	1.1350	0.104
H(6A)	4e	0.9924	0.4261	1.1693	0.104
H(8A)	4e	0.7794	0.5909	1.1377	0.092
H(9A)	4e	0.6043	0.6768	1.0548	0.083
H(10A)	4e	0.5755	0.5855	0.9419	0.063
H(13A)	4e	0.2334	0.4935	0.7366	0.102
H(13B)	4e	0.1960	0.6608	0.7117	0.102
H(14A)	4e	0.4047	0.7252	0.7449	0.075
H(16A)	4e	0.4048	−0.0366	0.9463	0.071
H(16B)	4e	0.3944	−0.2100	0.9270	0.071
H(17A)	4e	0.5423	−0.1810	0.8575	0.058

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cd(1)	4e	0.68671(2)	0.32016(2)	0.849006(8)	0.0378(1)	0.03411(9)	0.03279(9)	−0.00031(6)	0.00510(6)	−0.00047(6)
O(1)	4e	0.4701(2)	0.3936(2)	0.7979(1)	0.051(1)	0.059(1)	0.079(1)	−0.0089(9)	−0.001(1)	0.019(1)
O(2)	4e	0.6112(2)	0.5757(2)	0.7972(1)	0.053(1)	0.045(1)	0.062(1)	0.0012(8)	0.0078(9)	0.0059(8)
O(3)	4e	0.6461(2)	0.0573(2)	0.8208(1)	0.068(1)	0.041(1)	0.056(1)	−0.0099(8)	0.0292(9)	−0.0079(8)
O(4)	4e	0.5693(2)	0.1515(2)	0.9093(1)	0.068(1)	0.0368(9)	0.055(1)	−0.0042(8)	0.0240(9)	−0.0077(8)
O(5)	4e	0.7981(2)	0.3237(2)	0.7579(1)	0.087(1)	0.038(1)	0.053(1)	0.0087(9)	0.036(1)	0.0078(8)
C(1)	4e	0.9777(3)	0.1411(3)	0.8978(2)	0.048(1)	0.056(2)	0.069(2)	0.007(1)	0.020(1)	0.012(1)
C(2)	4e	1.0961(3)	0.0906(4)	0.9412(2)	0.043(2)	0.073(2)	0.105(3)	0.013(1)	0.019(2)	0.032(2)
C(3)	4e	1.1264(3)	0.1393(4)	1.0073(2)	0.035(1)	0.082(2)	0.110(3)	−0.001(1)	−0.007(2)	0.052(2)
C(4)	4e	1.0404(3)	0.2428(4)	1.0327(2)	0.043(1)	0.071(2)	0.063(2)	−0.017(1)	−0.011(1)	0.029(2)
C(5)	4e	1.0619(4)	0.2981(5)	1.1027(2)	0.070(2)	0.113(3)	0.060(2)	−0.032(2)	−0.029(2)	0.033(2)
C(6)	4e	0.9764(5)	0.3946(5)	1.1229(2)	0.101(3)	0.113(3)	0.037(2)	−0.052(3)	−0.011(2)	0.005(2)
C(7)	4e	0.8602(3)	0.4512(4)	1.0748(1)	0.079(2)	0.066(2)	0.036(1)	−0.036(2)	0.006(1)	−0.003(1)
C(8)	4e	0.7680(4)	0.5560(4)	1.0920(2)	0.112(3)	0.074(2)	0.052(2)	−0.045(2)	0.036(2)	−0.026(2)
C(9)	4e	0.6640(4)	0.6062(4)	1.0435(2)	0.091(2)	0.054(2)	0.073(2)	−0.021(2)	0.042(2)	−0.023(2)
C(10)	4e	0.6473(3)	0.5502(3)	0.9755(2)	0.060(2)	0.041(1)	0.061(2)	−0.006(1)	0.023(1)	−0.008(1)
C(11)	4e	0.8354(2)	0.3995(3)	1.0056(1)	0.049(1)	0.045(1)	0.034(1)	−0.020(1)	0.0053(9)	0.0023(9)
C(12)	4e	0.9255(2)	0.2911(3)	0.9842(1)	0.037(1)	0.049(1)	0.041(1)	−0.012(1)	−0.0025(9)	0.015(1)
C(13)	4e	0.2601(4)	0.5907(5)	0.7314(2)	0.060(2)	0.112(3)	0.075(2)	0.012(2)	−0.007(2)	0.019(2)
C(14)	4e	0.3819(3)	0.6270(4)	0.7509(2)	0.057(2)	0.065(2)	0.060(2)	0.013(1)	0.003(1)	0.010(1)
C(15)	4e	0.4950(3)	0.5238(3)	0.7836(1)	0.048(1)	0.049(1)	0.037(1)	0.011(1)	0.009(1)	0.010(1)
C(16)	4e	0.4308(3)	−0.1169(3)	0.9220(2)	0.057(2)	0.053(2)	0.072(2)	−0.006(1)	0.019(1)	0.006(1)
C(17)	4e	0.5181(3)	−0.0989(3)	0.8811(1)	0.059(2)	0.034(1)	0.055(2)	−0.003(1)	0.017(1)	−0.001(1)
C(18)	4e	0.5815(2)	0.0461(3)	0.8700(1)	0.040(1)	0.034(1)	0.043(1)	0.0022(9)	0.0055(9)	0.0020(9)
N(2)	4e	0.8959(2)	0.2396(2)	0.9181(1)	0.038(1)	0.044(1)	0.046(1)	0.0007(8)	0.0069(8)	0.0070(9)
N(1)	4e	0.7294(2)	0.4489(2)	0.9569(1)	0.045(1)	0.036(1)	0.038(1)	−0.0065(8)	0.0077(8)	−0.0018(8)

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References

1. Cai, T.-J.; Jiang, W.-J.; Peng, Z.-S.; Long, Y.-F.; Deng, Q.: Crystal structure of chloro(acrylato-*O,O'*)bis(1,10-phenanthroline-*N,N'*)cadmium(II), $CdCl(C_3H_3O_2)(C_{12}H_8N_2)_2$. Z. Kristallogr. NCS **221** (2006) 231-232.
2. Sen, S.; Saha, M. K.; Kundu, P.; Mitra, S.; Kruger, C.; Bruckmann, J.: Synthesis and structure of a heptacoordinated cadmium(II) complex. Inorg. Chim. Acta **288** (1999) 118-121.
3. Sheldrick, G. M.: Phase annealing in SHELX-90: direct methods for larger structures. Acta Crystallogr. A**46** (1990) 467-473.
4. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.