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Crystal structure of aqua-bis(acrylato-*O,O'*)(1,10-phenanthroline-*N,N'*)zinc(II) hydrate, $Zn(C_3H_3O_2)_2(H_2O)(C_{12}H_{10}N_2) \cdot H_2O$

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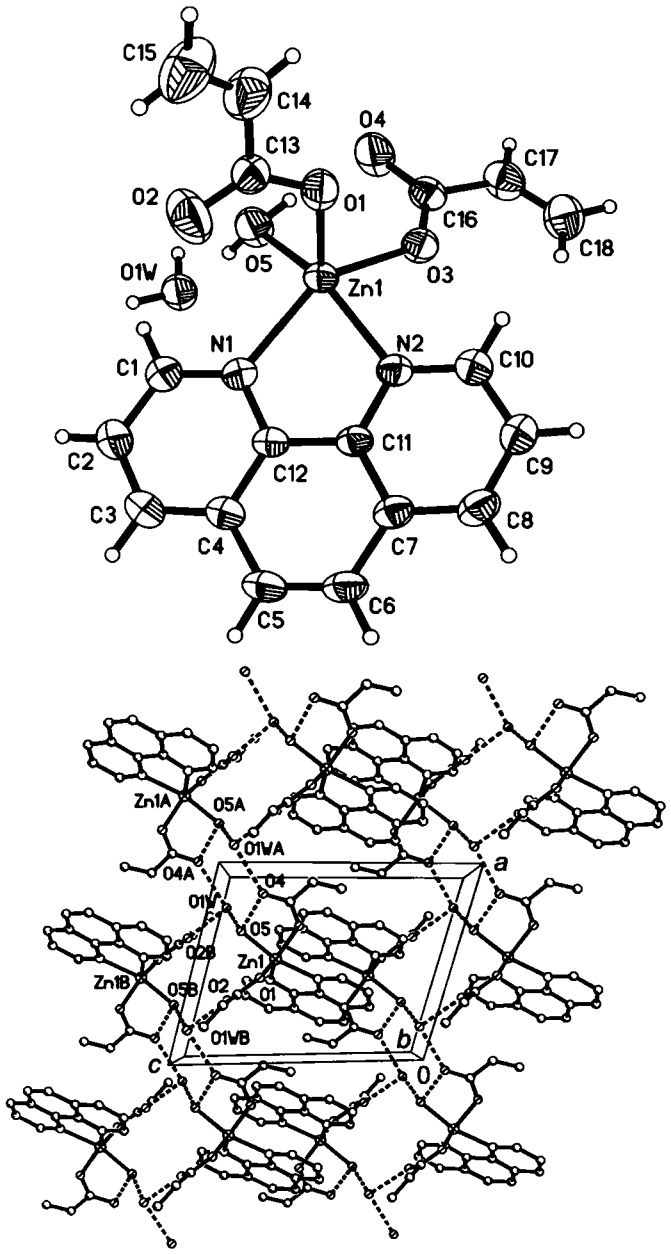

Abstract

$C_{18}H_{18}N_2O_6Zn$, triclinic, $P\bar{1}$ (no. 2), a = 9.2816(7) Å, b = 10.5205(8) Å, c = 11.0378(7) Å, α = 103.144(4)°, β = 99.428(4)°, γ = 111.743(3)°, V = 937.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.029, $wR_{ref}(F^2)$ = 0.080, T = 293 K.

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Source of material

A mixture solution of 18 ml H_2O/ethanol (1:1, *v/v*) containing ZnO (0.0814 g, 1.0 mmol), 1,10-phenanthroline monohydrate (0.198 g, 1.0 mmol) and acrylic acid (0.144 g, 2.0 mmol) was sealed in a Teflon-lined stainless steel vessel (25 ml), and the vessel was heated at 363 K for 7d, then cooled to room temperature and filtered off. The resultant filtrate was allowed to stand by slow evaporation at room temperature. The colorless block crystals generated 21 days later (yield 50 % based on the initial ZnO input).

Discussion

The title compound consists of $[Zn(C_3H_3O_2)_2(H_2O)(C_{12}H_{10}N_2)]$ complex molecules and lattice water molecules. The Zn atoms are each square pyramidally coordinated by two nitrogen atoms of one 1,10-phenanthroline ligand (N1, N2) and three oxygen atoms of aqua ligand (O5) and two monodentate acrylato anion ligands (O1, O3). Two crystallographically independent acrylato anions coordinate the Zn atoms in *syn* and *anti* modes, respectively. The apex is occupied by one acrylato O1 atom, and the Zn atom is shifted by 0.554 Å from the basal plane defined by atoms O3, O5, N1 and N2 (figure, top). This differs from that observed in the previously reported structure of aquaphenanthrolinebis(4-nitrobenzoato)zinc(II), $[Zn(C_7H_4NO_4)_2(C_{12}H_8N_2)(H_2O)]$, where the five-coordinated Zn(II) atom exhibits a distorted trigonal bipyramidal environment with one aromatic pyridyl N residing at the basal positions and the other locating at the axial site [1]. The apical Zn1—O1 distance (1.956(1) Å) is shorter than the basal Zn—O3 lengths (2.049(1) Å), and the Zn1—O1 and Zn1—O3 distances are according with those observed in some monodentate carboxyl zinc complexes containing N-donor ligands [1-4]. The Zn1—O5 distance is 2.067(1) Å, which is slightly longer than that (2.043(2) Å) in the related complex [1]. The Zn1—N1, Zn1—N2 bond lengths are 2.140(1) Å and 2.148(1) Å, respectively, which are in the region of Zn—N bond distances (2.089(3) Å – 2.174(3) Å) in $[Zn(C_7H_4NO_4)_2(C_{12}H_8N_2)(H_2O)]$ [1]. The *cis* bond angles at the central Zn atom from the apical O1 atom fall in the range of 97.83(6)° – 120.25(6)°, indicating a significant deviation from the value for a perfect square pyramidal environment. Within the crystal structure, there exist strong intra-molecular hydrogen-bonding interactions and intermolecular hydrogen-bonding interactions. On the one hand, the coordinated aqua molecules donor hydrogen atoms to the uncoordinated acrylato oxygen atoms (O4) (d(O···O) = 2.631 Å, ∠O–H···O = 159.9°) and lattice water molecules oxygen atoms (O1w) (d(O···O) = 2.697 Å, ∠O–H···O = 173.85°) to form strong intramolecular hydrogen bonds. On the other hand, the lattice water molecules functioning as hydrogen-bond donors contribute hydrogen atoms to the uncoordinated acrylato oxygen atoms ($O2^i$, $O4^{ii}$) (d(O···$O2^i$) = 2.695 Å, ∠O–H···$O2^i$ = 172.2°, d(O···$O4^{ii}$) = 2.699 Å, ∠O–H···O^{ii} = 173.6°)

of neighboring complex molecules to form strong intermolecular hydrogen bonds (i: $-x+1,-y+2,-z+2$; ii: $-x+2,-y+2,-z+2$) (figure, bottom). Along [100] direction the complex and water molecules are interlinked by intra- and intermolecular hydrogen bonds into one-dimensional double chains, which contain unusual six-membered chair-like conformation water-oxygen cycle (figure, bottom). The resulting one-dimensional double chains are further held by π-π interactions between the 1,10-phenanthroline ligands of neighboring double chains into layers (mean interplanar distance: 3.546(7) Å).

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.32 × 0.40 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	13.47 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD II, ω
$2\theta_{max}$:	56.78°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11876, 4634
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4051
$N(param)_{refined}$:	244
Programs:	SHELXS-97 [5], SHELXL-97 [6]

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

Atom	Site	x	y	z	U_{iso}
H(5B)	2*i*	0.7867	0.8809	0.8887	0.101
H(5A)	2*i*	0.7454	1.0012	0.9205	0.077
H(2W)	2*i*	0.9248	1.2001	1.0541	0.073
H(1W)	2*i*	0.7808	1.2250	1.0339	0.105
H(1A)	2*i*	0.4845	1.0213	0.8987	0.068
H(8A)	2*i*	0.2219	0.6752	0.1743	0.068
H(3A)	2*i*	0.2129	1.1496	0.6826	0.070
H(14A)	2*i*	0.3484	0.4081	0.8557	0.107
H(2A)	2*i*	0.3449	1.1615	0.8837	0.075
H(17A)	2*i*	1.0491	0.6957	0.6894	0.079
H(18A)	2*i*	0.8213	0.6373	0.4680	0.095
H(18B)	2*i*	0.9835	0.6126	0.4769	0.095
H(10A)	2*i*	0.4907	0.5770	0.4251	0.061
H(9A)	2*i*	0.3582	0.5389	0.2150	0.069
H(6A)	2*i*	0.1392	0.8775	0.2642	0.068
H(5C)	2*i*	0.1360	1.0381	0.4373	0.067
H(15B)	2*i*	0.1290	0.4898	0.9390	0.131
H(15C)	2*i*	0.1569	0.3493	0.9470	0.131

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}
Zn(1)	2*i*	0.54492(2)	0.77682(2)	0.71164(2)	0.0477(1)	0.0501(1)	0.0401(1)	0.02773(9)	0.00777(8)	0.01563(8)
O(1)	2*i*	0.4423(2)	0.5982(2)	0.7519(1)	0.081(1)	0.0563(8)	0.0625(8)	0.0341(7)	0.0347(7)	0.0228(6)
O(5)	2*i*	0.7033(2)	0.9043(2)	0.8915(1)	0.0613(8)	0.0711(9)	0.0533(7)	0.0369(7)	−0.0012(6)	0.0043(6)
O(1W)	2*i*	0.8438(2)	1.1942(2)	0.9930(1)	0.0589(8)	0.0766(9)	0.0498(7)	0.0386(7)	0.0043(6)	0.0108(6)
O(3)	2*i*	0.7238(2)	0.7354(2)	0.6491(1)	0.0493(7)	0.0752(9)	0.0492(7)	0.0369(6)	0.0107(5)	0.0209(6)
O(4)	2*i*	0.8989(2)	0.7858(2)	0.8360(2)	0.073(1)	0.108(1)	0.0610(9)	0.061(1)	−0.0056(7)	0.0056(8)
O(2)	2*i*	0.3207(3)	0.6990(2)	0.8662(2)	0.119(2)	0.095(1)	0.154(2)	0.071(1)	0.093(2)	0.062(1)
N(2)	2*i*	0.4327(2)	0.7281(2)	0.5103(1)	0.0425(7)	0.0476(7)	0.0419(7)	0.0221(6)	0.0085(5)	0.0165(6)
N(1)	2*i*	0.4329(2)	0.9220(2)	0.7146(1)	0.0484(7)	0.0504(8)	0.0404(7)	0.0242(6)	0.0102(6)	0.0170(6)
C(1)	2*i*	0.4291(2)	1.0138(2)	0.8173(2)	0.067(1)	0.062(1)	0.0460(9)	0.035(1)	0.0134(8)	0.0168(8)
C(8)	2*i*	0.2746(2)	0.6892(2)	0.2588(2)	0.059(1)	0.069(1)	0.0400(8)	0.0263(9)	0.0056(7)	0.0182(8)
C(3)	2*i*	0.2672(2)	1.0918(2)	0.6899(2)	0.062(1)	0.056(1)	0.069(1)	0.0360(9)	0.0184(9)	0.0216(9)
C(14)	2*i*	0.2964(4)	0.4683(3)	0.8696(3)	0.118(2)	0.069(2)	0.086(2)	0.031(2)	0.051(2)	0.031(1)
C(2)	2*i*	0.3462(3)	1.0998(2)	0.8091(2)	0.075(1)	0.061(1)	0.057(1)	0.038(1)	0.020(1)	0.0136(9)
C(17)	2*i*	0.9537(2)	0.6915(3)	0.6423(2)	0.050(1)	0.078(1)	0.080(1)	0.035(1)	0.020(1)	0.027(1)
C(18)	2*i*	0.9160(3)	0.6425(3)	0.5173(3)	0.073(2)	0.097(2)	0.085(2)	0.046(1)	0.038(1)	0.031(1)
C(11)	2*i*	0.3521(2)	0.8082(2)	0.4868(2)	0.0353(7)	0.0454(8)	0.0413(7)	0.0169(6)	0.0094(6)	0.0187(6)
C(12)	2*i*	0.3518(2)	0.9119(2)	0.5966(2)	0.0363(7)	0.0417(8)	0.0441(8)	0.0162(6)	0.0095(6)	0.0191(6)
C(4)	2*i*	0.2680(2)	0.9966(2)	0.5788(2)	0.0427(8)	0.0473(9)	0.0558(9)	0.0215(7)	0.0127(7)	0.0227(7)
C(16)	2*i*	0.8526(2)	0.7416(2)	0.7145(2)	0.0462(9)	0.056(1)	0.058(1)	0.0266(8)	0.0101(8)	0.0189(8)
C(13)	2*i*	0.3529(2)	0.5985(2)	0.8265(2)	0.054(1)	0.056(1)	0.0492(9)	0.0246(8)	0.0139(8)	0.0160(8)
C(7)	2*i*	0.2713(2)	0.7932(2)	0.3620(2)	0.0409(8)	0.057(1)	0.0428(8)	0.0197(7)	0.0072(6)	0.0211(7)
C(10)	2*i*	0.4345(2)	0.6314(2)	0.4098(2)	0.054(1)	0.054(1)	0.0489(9)	0.0282(8)	0.0125(8)	0.0159(8)
C(9)	2*i*	0.3557(2)	0.6084(2)	0.2828(2)	0.065(1)	0.062(1)	0.0439(9)	0.0286(9)	0.0128(8)	0.0118(8)
C(6)	2*i*	0.1909(2)	0.8849(2)	0.3469(2)	0.053(1)	0.071(1)	0.053(1)	0.0323(9)	0.0060(8)	0.0306(9)
C(5)	2*i*	0.1893(2)	0.9808(2)	0.4500(2)	0.053(1)	0.063(1)	0.063(1)	0.0329(9)	0.0109(8)	0.0316(9)
C(15)	2*i*	0.1844(4)	0.4327(3)	0.9232(3)	0.122(2)	0.087(2)	0.091(2)	0.007(2)	0.050(2)	0.026(2)

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