Crystal structure of diaqua-bis(3-oxo-3,4-dihydroquinoxaline-2-carboxylato-κ^2*N*,*O*)- zinc(II)-methanol-water (1:1:1), $[Zn(C_9H_5N_2O_3)_2(H_2O)_2]\cdot CH_3OH\cdot H_2O$, $C_{19}H_{20}N_4O_{10}Zn$

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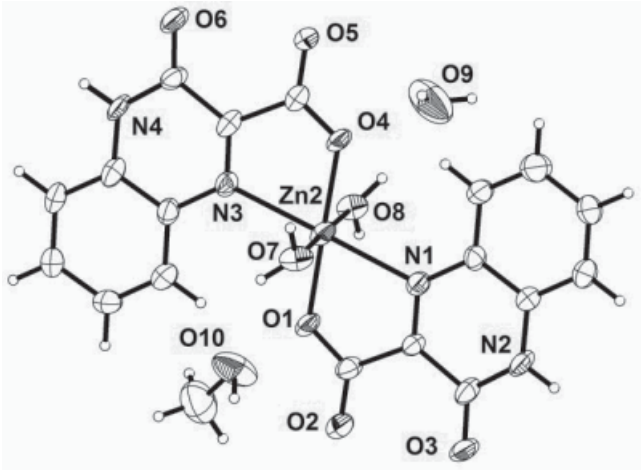

Abstract

$C_{19}H_{20}N_4O_{10}Zn$, monoclinic, $P2_1$ (no. 4), a = 7.0535(5) Å, b = 16.181(1) Å, c = 9.4852(5) Å, β = 101.091(6)°, V = 1062.3 Å^3, Z = 2, $R_{gt}(F)$ = 0.0485, $wR_{ref}(F^2)$ = 0.1281, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.15×0.18×0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.23 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ and ω
$2\theta_{max}$:	52.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4308, 2871
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2550
$N(param)_{refined}$:	308
Programs:	SHELX [9]

Source of material

The title compound was synthesized by adding the CH_3OH solution (6 mL) of 3-hydroxy-2-quinoxalinecarboxylic acid (0.5 mmol) dropwise to a stirred water solution (4 mL) of $Zn(CH_3COO)_2\cdot 2H_2O$ (0.5 mmol) at 298 K. Then the reaction mixture was filtered and the filtrate stood for about two weeks until the colourless block-shaped crystals were obtained. The crystals suitable for X-ray diffraction were collected by filtration, washed with water and ethanol and dried in air (47 % yield based on Zn). **Elemental analysis** calcd. for ($C_{19}H_{20}N_4O_{10}Zn$): H 3.81, C 43.08, N 10.58, O 30.20 %; found: H 3.72, C 43.15, N 10.52, O 30.25 %.

Experimental details

All the hydrogen atoms were included in calculated positions and treated as riding on the parent atom with d(O–H) = 0.85 Å, d(C–H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C,O).

Discussion

The research in coordination chemistry has grown progressively during the last decades. The potential application of hybrid ligands which permits to connect organic molecules to metals by two different heteroatoms (N, S, O, P, etc.) to form metallocycles has focused much interest. It is well known that the relationship between the steric and electronic preferences to metal ion and the coordination properties of the ligand plays an important role in the structure of the compounds. In this context, aromatic carboxylate ligands, and especially N-heterocyclic carboxylates, have been intensively studied for their versatile coordination modes and potential applicability to different fields, pharmaceutical, catalyst and magnetic properties and others. These studies are extensively reported in the literature [1-8]. As a part of our continuing investigations on inorganic-organic hybrid compounds containing zinccarboxylate systems, the title complex was synthesized. The crystal structure of the title complex consists of the mononuclear zinc(II) unit $[Zn(C_9H_5N_2O_3)_2(H_2O)_2]$, one free methanol molecule and one lattice water molecule. The zinc(II) atom is located on an inversion center and is six-coordinated by two carboxylate oxygen atoms and two N donors from two different 3-oxo-3,4-dihydroquinoxaline-2-carboxylato ligands, and two O atoms from two coordinating water molecules, forming a distorted octahedron. N1, N3, O1 and O4 atoms constitute the basal plane of the octahedron (the mean deviation from plane of 0.0522 Å). The O7 and O8 atoms occupy the two axial positions. The axial Zn–O7 and Zn–O8 distances are 2.074(5) and 2.082(5) Å, respectively. The basal Zn–O (2.006(4), 2.018(5) Å) and Zn–N (2.263(5), 2.270(5) Å) bond lengths are in normal range. The bond angles around the Zn(II) are in the range of 76.11(18)-178.5(2)°. All the molecules, ligands and solvate molecules participate in intermolecular N–H···O and O–H···O hydrogen bonds, which further stabilize the 3D framework.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1W)	2*a*	0.1195	0.8477	0.5456	0.066
H(2W)	2*a*	–0.0338	0.8015	0.4816	0.066
H(4W)	2*a*	0.5046	0.6011	0.7372	0.060
H(3W)	2*a*	0.3313	0.5595	0.6789	0.060
H(5W)	2*a*	0.1094	0.4837	0.6066	0.151
H(6W)	2*a*	0.2332	0.4174	0.5969	0.151
H(10D)	2*a*	0.2375	0.9973	0.4406	0.114
H(2)	2*a*	0.5660	0.6708	0.0917	0.044
H(4D)	2*a*	–0.1328	0.7641	1.0993	0.043
H(4)	2*a*	0.3298	0.5759	–0.0314	0.052
H(5)	2*a*	0.0588	0.5005	–0.0277	0.060
H(6)	2*a*	–0.0961	0.5082	0.1700	0.068
H(7A)	2*a*	0.0323	0.5888	0.3628	0.056
H(13)	2*a*	0.1044	0.8589	1.2217	0.05
H(14)	2*a*	0.3843	0.9316	1.2181	0.054
H(15)	2*a*	0.5473	0.9140	1.0288	0.057
H(16)	2*a*	0.4283	0.8264	0.8439	0.048
H(19A)	2*a*	0.3502	1.0388	0.6331	0.122
H(19B)	2*a*	0.4987	0.9824	0.5749	0.122
H(19C)	2*a*	0.3839	0.9476	0.6881	0.122

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(2)	2*a*	0.2356(1)	0.70563(6)	0.60799(6)	0.0356(3)	0.0416(4)	0.0275(3)	–0.0069(4)	0.0169(2)	–0.0022(3)
O(1)	2*a*	0.4827(6)	0.7697(3)	0.6343(4)	0.037(3)	0.049(3)	0.026(2)	–0.009(2)	0.017(2)	–0.003(2)
O(2)	2*a*	0.7078(7)	0.8230(4)	0.5325(5)	0.053(3)	0.075(4)	0.058(3)	–0.029(3)	0.035(3)	–0.028(3)
O(3)	2*a*	0.7404(7)	0.7648(4)	0.2682(5)	0.046(3)	0.083(4)	0.037(2)	–0.033(3)	0.025(2)	–0.019(3)
O(4)	2*a*	–0.0087(6)	0.6373(3)	0.5861(4)	0.040(3)	0.055(3)	0.027(2)	–0.016(2)	0.021(2)	–0.008(2)
O(5)	2*a*	–0.2250(7)	0.5815(3)	0.6958(5)	0.057(3)	0.056(3)	0.051(3)	–0.029(3)	0.036(2)	–0.023(3)
O(6)	2*a*	–0.3044(7)	0.6667(3)	0.9292(4)	0.038(3)	0.077(4)	0.032(2)	–0.019(3)	0.019(2)	–0.013(2)
O(7)	2*a*	0.0889(6)	0.8036(3)	0.4982(5)	0.032(2)	0.049(3)	0.051(3)	–0.006(2)	0.010(2)	0.007(2)
O(8)	2*a*	0.3822(7)	0.6043(3)	0.7146(4)	0.033(2)	0.044(3)	0.044(2)	–0.003(2)	0.010(2)	0.005(2)
O(9)	2*a*	0.207(1)	0.4565(4)	0.650(1)	0.064(4)	0.061(5)	0.172(7)	0.003(4)	0.007(5)	–0.032(5)
O(10)	2*a*	0.233(1)	0.9569(4)	0.4972(6)	0.094(5)	0.049(3)	0.079(4)	–0.023(4)	0.000(4)	0.016(3)
N(1)	2*a*	0.3430(7)	0.6810(3)	0.4011(5)	0.029(3)	0.037(3)	0.027(2)	–0.003(2)	0.009(2)	–0.003(2)
N(2)	2*a*	0.5130(7)	0.6734(3)	0.1660(5)	0.032(3)	0.054(4)	0.028(2)	–0.002(3)	0.017(2)	–0.003(2)
N(3)	2*a*	0.1164(8)	0.7358(3)	0.8065(5)	0.040(3)	0.031(3)	0.024(2)	0.000(2)	0.013(2)	0.001(2)
N(4)	2*a*	–0.0731(7)	0.7568(4)	1.0293(5)	0.030(3)	0.058(4)	0.024(2)	–0.004(3)	0.017(2)	–0.008(2)
C(1)	2*a*	0.4954(8)	0.7243(3)	0.3990(5)	0.025(3)	0.032(4)	0.024(2)	–0.001(3)	0.009(2)	0.001(2)
C(2)	2*a*	0.5972(9)	0.7230(5)	0.2755(6)	0.036(3)	0.050(5)	0.025(3)	0.000(3)	0.014(2)	0.000(3)
C(3)	2*a*	0.3511(9)	0.6275(4)	0.1647(6)	0.033(3)	0.034(4)	0.030(3)	–0.002(3)	0.012(3)	0.000(3)
C(4)	2*a*	0.271(1)	0.5783(5)	0.0484(6)	0.056(5)	0.051(4)	0.025(3)	–0.009(4)	0.016(3)	–0.006(3)
C(5)	2*a*	0.110(1)	0.5340(5)	0.0501(7)	0.059(5)	0.059(5)	0.035(4)	–0.022(4)	0.014(3)	–0.020(3)
C(6)	2*a*	0.016(1)	0.5382(6)	0.1698(7)	0.062(5)	0.073(6)	0.037(4)	–0.029(5)	0.014(3)	–0.013(4)
C(7)	2*a*	0.093(1)	0.5862(5)	0.2841(7)	0.047(4)	0.059(5)	0.038(3)	–0.022(4)	0.021(3)	–0.015(3)
C(8)	2*a*	0.2617(9)	0.6316(4)	0.2847(5)	0.038(3)	0.029(3)	0.020(2)	–0.002(3)	0.007(2)	–0.001(2)
C(9)	2*a*	0.5705(8)	0.7770(4)	0.5311(6)	0.024(3)	0.043(4)	0.030(3)	–0.005(3)	0.008(2)	0.000(3)
C(10)	2*a*	–0.0394(8)	0.6959(5)	0.8088(5)	0.031(3)	0.046(4)	0.027(2)	0.005(4)	0.013(2)	0.002(3)
C(11)	2*a*	–0.1522(8)	0.7028(6)	0.9249(5)	0.033(3)	0.047(3)	0.024(2)	–0.008(5)	0.011(2)	0.002(4)
C(12)	2*a*	0.0946(9)	0.8006(5)	1.0323(6)	0.036(3)	0.049(4)	0.023(3)	0.006(3)	0.010(2)	–0.001(3)
C(13)	2*a*	0.168(1)	0.8528(5)	1.1451(7)	0.049(4)	0.050(4)	0.032(3)	–0.001(4)	0.020(3)	–0.012(3)
C(14)	2*a*	0.336(1)	0.8956(5)	1.1434(7)	0.052(5)	0.047(4)	0.042(4)	–0.008(4)	0.019(3)	–0.017(3)
C(15)	2*a*	0.433(1)	0.8852(5)	1.0292(7)	0.046(4)	0.053(5)	0.046(4)	–0.022(4)	0.018(3)	–0.010(3)
C(16)	2*a*	0.362(1)	0.8333(4)	0.9190(7)	0.045(4)	0.046(4)	0.033(3)	–0.005(3)	0.018(3)	–0.001(3)
C(17)	2*a*	0.1910(9)	0.7904(4)	0.9179(6)	0.040(3)	0.032(3)	0.026(3)	0.002(3)	0.012(3)	–0.002(2)
C(18)	2*a*	–0.0988(9)	0.6331(4)	0.6868(6)	0.034(3)	0.038(4)	0.029(3)	0.003(3)	0.013(2)	0.000(3)
C(19)	2*a*	0.377(2)	0.9835(6)	0.607(1)	0.115(9)	0.068(7)	0.059(5)	–0.011(7)	0.009(6)	–0.008(5)

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