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The crystal structure of bis(*N*-oxy-2-(1*H*-tetrazol-1-yl) acetamide $\kappa^2 O, O'$)-diaqua-zinc(II), $C_6H_{12}ZnN_{10}O_6$

Table 1: Data collection and handling.

Crystal:	Light red plate
Size:	0.60 × 0.23 × 0.07 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.89 mm ⁻¹
Diffractometer, scan mode:	Rigaku Oxford Diffraction
Enhance, ω	
$\theta_{\max}$, completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5714, 1366, 0.030
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1147
$N(\text{param})_{\text{refined}}$:	108
Programs:	CrysAlis ^{PRO} [1], SHELX [2], Diamond [3]

<https://doi.org/10.1515/ncrs-2019-0856>

Received November 24, 2019; accepted January 28, 2020; available online March 11, 2020

Abstract

[Zn(C₃H₄N₅O₂)₂(H₂O)₂], orthorhombic, *Pbcn* (no. 60), $a = 19.6175(12)$ Å, $b = 7.2887(5)$ Å, $c = 9.3499(6)$ Å, $V = 1336.9(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0276$, $wR_{\text{ref}}(F^2) = 0.0605$, $T = 293(2)$ K.

CCDC no.: 1961562

The molecular 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.

Source of material

KOH (1.68 g, 0.03 mol) was added to a solution of hydroxylamine hydrochloride (1.39 g, 0.02 mol) in MeOH (20 mL) at 0 °C. The mixture was stirred at 0 °C for 2 h. The suspension was filtered. The filtrate was added to a solution of methyl 2-(1*H*-tetrazol-1-yl) acetate (1.42 g, 0.01 mol) in MeOH (10 mL) at 0 °C. The solution was then stirred at 0 °C for 1 h.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.0000	0.19646(5)	0.2500	0.01940(12)
N1	0.19127(9)	0.6455(2)	0.18409(19)	0.0219(4)
N2	0.19539(11)	0.6983(3)	0.3211(2)	0.0342(5)
N3	0.25857(12)	0.6778(3)	0.3575(2)	0.0396(6)
N4	0.29562(11)	0.6119(3)	0.2478(2)	0.0372(5)
N5	0.06750(9)	0.3798(3)	0.02643(18)	0.0211(4)
H5A	0.0775	0.4126	−0.0648	0.026(6)*
O1	0.07560(8)	0.4192(2)	0.26430(13)	0.0239(4)
O2	0.03054(9)	0.2226(2)	0.04139(16)	0.0261(4)
O1W	−0.06656(8)	−0.0069(2)	0.18290(18)	0.0310(4)
H1WA	−0.0609	−0.0655	0.1045	0.057(10)*
H1WB	−0.1112	0.0188	0.1791	0.071(11)*
C1	0.25247(12)	0.5937(4)	0.1422(3)	0.0319(6)
H1A	0.2633	0.5508	0.0514	0.038*
C2	0.12583(11)	0.6467(3)	0.1084(2)	0.0225(5)
H2A	0.1336	0.6590	0.0064	0.027*
H2B	0.0988	0.7506	0.1399	0.027*
C3	0.08790(10)	0.4716(3)	0.1378(2)	0.0183(5)

The resulting solution was filtered and the solid was washed with EtOH (3 × 10 mL), dried under vacuum pressure to afford 770 mg (crude) of potassium *N*-oxy-2-(1*H*-tetrazol-1-yl) acetamide.

A solution of Zn(NO₃)₂ · H₂O (148.7 mg, 0.5 mmol) in DMF (3 mL) was added dropwise to a stirred suspension of potassium *N*-oxy-2-(1*H*-tetrazol-1-yl) acetamide (90.6 mg,

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0.5 mmol) in DMF (3 mL); 0.5 M HCl (1 mL) was then added to the mixture at room temperature. The suspension was stirred at room temperature for 16 h. The mixture was filtered. After five months, light red plate crystals of the title complex were obtained from the filtrate by slow evaporation at room temperature.

Experimental details

All the H atoms attached to carbons were placed at calculated positions (0.93 Å and 0.97 Å) and included in the refinement using the riding model approximation, with $U_{\text{iso}}(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$. The hydrogen atoms of O—H and N—H were located from the difference Fourier map and their positions and U_{iso} parameters were initially freely refined and the positions subsequently constrained using the SHELXL AFIX 3 command.

Comment

Supramolecular chemistry is particularly attractive to researchers due to potential application in electro-photics [4], catalysts [5], molecular recognition [6] and healthcare [7] etc. By tuning supramolecular interactions such as hydrogen bonding, guest-host interactions and π – π stacking, material properties can be profoundly altered. In this paper, we have chosen *N*-oxy-2-(1*H*-tetrazol-1-yl) acetamide as the ligand, with the carbonyl/oxime groups coordinating to Zn(II) to construct a supramolecular complex.

As shown in the figure (unlabelled atoms are related by the symmetry operation a: $-x, y, -z + 1/2$), the title complex contains one Zn(II) atom, two *N*-oxy-2-(1*H*-tetrazol-1-yl) acetamide ligands and two water molecules. The zinc center is six-coordinated by two carbonyl O atoms [O(1), O(1a)], two oxime O atoms [O(2), O(2a)] and two water O atoms [O(1W), O(1Wa)] related by a symmetry operation given above, forming a distorted ZnO₆ octahedral coordination geometry. The Zn(II)–O bond lengths are 2.203(2) Å (carbonyl), 2.049(2) Å (oxime) and 2.072(2) Å (water), respectively. The Zn–O bond length [2.203(2) Å, carbonyl] is slightly longer than the corresponding Zn(II)–O(carbonyl) (2.189 Å) in [Zn(OAcF)(BA)(Im)₂] (BA = benzohydroxamate) [8]. The Zn(II)–O bond length [2.049(2) Å, oxime] is identical to the bond length of 2.050 Å in [Zn₂(OAcF)₃(tmen)₂(AA)] (tmen = *N,N,N',N'*-tetramethylethylenediamine; AA = acetohydroxamate) [8]. Meanwhile, the Zn(II)–O bond length [2.072(2) Å, water] is between the bond lengths [2.052(8) and 2.087(2) Å] in [(1')₂Zn₈Na₂(H₂O)₂₁] · 20H₂O [9] and [Zn(L)(H₂O)] · H₂O [10]. Moreover, the five-membered ring composed of Zn(1), O(1), C(3), N(5) and O(2), is almost coplanar with r.m.s deviation of 0.0785 Å. We note that the structures of other related compounds have also been reported in the literature [11, 12].

In the complex, there are intermolecular hydrogen bonds, connecting adjacent complexes (as indicated in the second figure, (N(5)–H(5)···O(1b); O(1W)–H(1WA)···O(2c) and O(1W)–H(1WB)···N(4d) symmetry codes: b: $x, -y + 1, z - 1/2$; c: $-x, -y, -z$; d: $x - 1/2, y - 1/2, -z + 1/2$). The distances of N(5)···O(1b), O(1W)···O(2c) and O(1W)···N(4d) are 2.860(2), 2.715(2) and 2.912(3) Å, respectively; the angles of (N(5)–H(5)···O(1b), O(1W)–H(1WA)···O(2c) and O(1W)–H(1WB)···N(4d) are 154.39, 165.47 and 156.91°, respectively. As a result, the desired complex exhibits a three-dimensional supramolecular network.

Acknowledgements: We gratefully acknowledge financial supports by the National Natural Science Foundation of China (no. 21771066) and the Natural Science Foundation of Fujian Province of China (No. 2015H0024).

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