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Crystal structure of bis(acetato- κ^1O)-bis(1-(pyridin-2-yl)ethan-1-one oxime- κ^2N,N')zinc(II), $C_{18}H_{22}N_4O_6Zn$

Table 1: Crystal collection and handling.

Crystal:	Block, colorless
Size:	0.47 × 0.41 × 0.36 mm
Wavelength:	Mo K α radiation ($\lambda = 0.71073$ Å)
μ :	1.244 mm $^{-1}$
Diffractometer, scan mode:	APEX2, Φ and ω -scans
$\theta_{\max}$, completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11215, 4165, 0.0368
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2844
$N(\text{param})_{\text{refined}}$:	268
Programs:	Bruker programs [1], SHELX [2, 3]

Source of materials

Hydroxylamine hydrochloride (0.695 g, 10 mmol) was added to sodium hydroxide (0.401 g, 10 mmol) in deionized water (10 mL). The solution was added dropwise to 2-acetylpyridine (1.211 g, 10 mmol) in methanol (10 mL). The reaction was stirred for 4 h. A solution of zinc acetate dihydrate (2.195 g, 10 mmol) in MeOH (10 mL) was added. The resulting solution was stirred at 25 °C for about 4 h and was then allowed to slowly concentrate by solvent evaporation at room temperature. Colourless block crystals were obtained within a week. The elemental analysis calculated for $C_{18}H_{22}N_4O_6Zn$: C 47.43, H 4.86, N 12.29%; found: C 47.61, H 5.96, N 12.05%.

Experimental details

All hydrogen atoms were placed geometrically and treated as riding on their parent atoms with O–H 0.82 Å, C–H 0.96 Å [$U_{\text{iso}}(\text{H}) = 1.5$ times U_{eq} (O, water), (C, methyl)] and C–H 0.93 Å [$U_{\text{iso}}(\text{H}) = 1.2$ times U_{eq} (C, aromatic ring)].

Comment

Pyridyl oximes have the general formula (py)C(R)NOH, where py is a pyridyl group attached to the oxime carbon atom and R can be a donor or a non-donor group [4]. Pyridyl oxime ligands represent one of the most widely utilized classes of ligands in coordination chemistry [5]. These complexes have been used in the fields of magnetism, catalysis and enzymatic reactions [6–8].

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Abstract

$C_{18}H_{22}N_4O_6Zn$, monoclinic, $P2_1/c$ (no. 14), $a = 9.9422(18)$ Å, $b = 16.904(3)$ Å, $c = 15.5509(19)$ Å, $\beta = 128.569(7)^\circ$, $V = 2043.4(6)$ Å 3 , $Z = 4$, $R_{\text{gt}}(F) = 0.0347$, $wR_{\text{ref}}(F^2) = 0.0663$, $T = 293(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Zn1	0.24610(3)	0.30394(2)	0.36336(2)	0.03623(10)
O1	0.3546(2)	0.31736(11)	0.60034(13)	0.0531(5)
H1	0.264696	0.342576	0.561296	0.080
O2	0.1161(2)	0.33042(11)	0.12196(13)	0.0498(5)
H6	0.194076	0.362632	0.158898	0.075
O3	0.3599(2)	0.42701(11)	0.24287(15)	0.0563(5)
O4	0.4415(2)	0.37363(11)	0.39884(14)	0.0538(5)
O5	0.0773(2)	0.39212(11)	0.47065(17)	0.0604(5)
O6	0.0959(2)	0.39836(11)	0.33607(15)	0.0566(5)
N1	0.4208(2)	0.19967(12)	0.40698(16)	0.0390(5)
N2	0.3817(3)	0.27778(12)	0.53567(16)	0.0390(5)
N3	0.0523(2)	0.21472(12)	0.31748(16)	0.0384(5)
N4	0.0949(2)	0.28907(12)	0.18851(15)	0.0359(5)
C1	0.5243(3)	0.17981(15)	0.5143(2)	0.0420(6)
C2	0.6426(4)	0.11974(18)	0.5540(3)	0.0670(9)
H2	0.714847	0.107645	0.628600	0.080
C3	0.6537(4)	0.0775(2)	0.4829(3)	0.0806(10)
H3	0.732366	0.036421	0.508805	0.097
C4	0.5474(4)	0.09695(19)	0.3738(3)	0.0676(9)
H4	0.551344	0.069063	0.323866	0.081
C5	0.4343(3)	0.15892(17)	0.3398(2)	0.0506(7)
H5	0.363840	0.172908	0.265795	0.061
C6	0.4992(3)	0.22471(16)	0.5851(2)	0.0414(6)
C7	0.6013(4)	0.20585(18)	0.7048(2)	0.0641(8)
H7A	0.592469	0.248866	0.741318	0.096
H7B	0.719737	0.198116	0.736621	0.096
H7C	0.557219	0.158482	0.713154	0.096
C8	−0.0551(3)	0.19628(15)	0.2098(2)	0.0372(6)
C9	−0.1791(4)	0.13878(18)	0.1701(3)	0.0615(8)
H9	−0.256333	0.128139	0.095156	0.074
C10	−0.1869(4)	0.09742(19)	0.2428(3)	0.0777(10)
H10	−0.267386	0.057183	0.217561	0.093
C11	−0.0765(4)	0.11540(18)	0.3524(3)	0.0664(9)
H11	−0.079899	0.087764	0.402693	0.080
C12	0.0394(3)	0.17531(16)	0.3859(2)	0.0503(7)
H12	0.112453	0.189005	0.459999	0.060
C13	−0.0314(3)	0.24047(15)	0.13785(18)	0.0357(6)
C14	−0.1450(3)	0.22688(16)	0.01657(19)	0.0514(7)
H14A	−0.154104	0.274846	−0.019798	0.077
H14B	−0.257202	0.211092	−0.008894	0.077
H14C	−0.096771	0.185966	0.000414	0.077
C15	0.6036(3)	0.48013(16)	0.4097(2)	0.0583(8)
H15A	0.709376	0.453568	0.438357	0.087
H15B	0.609405	0.500815	0.469414	0.087
H15C	0.585817	0.522745	0.362774	0.087
C16	0.4567(3)	0.42255(14)	0.3447(2)	0.0373(6)
C17	−0.0897(3)	0.48876(16)	0.3320(2)	0.0610(8)
H17A	−0.202907	0.468916	0.298678	0.091
H17B	−0.088525	0.513465	0.276829	0.091
H17C	−0.058312	0.526861	0.387473	0.091
C18	0.0372(3)	0.42130(16)	0.3837(2)	0.0454(7)

The asymmetric unit of the title structure contains one complete complex (*cf.* the figure). The complex consists of two 1-(pyridin-2-yl)ethan-1-one oxime, zinc and two acetate ligands. The Zn atom is hexa coordinated in a

distorted octahedral coordination geometry, defined by two O atoms from the acetate ligands and four N atoms from the (py)C(Me)NOH ligands. The basal plane is formed by the atoms N1, N2, N4 and O6, the axial positions are occupied by N3 and O4. The Zn–N bond lengths range from 2.149(19) Å to 2.261(2) Å and the Zn–O bond lengths are 2.032(17) Å, 2.041(18) Å respectively. The bond angles at the Zn atom are in the range of 72.57(7)° to 175.70(7)°. Bond lengths and angles are in the expected ranges [9–11]. Intramolecular hydrogen bonds are present (*cf.* the figure).

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