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Crystal structure of bis-[*N*-(1-pyrazin-2-yl-ethylidene)-cyanoacetic acid-hydrazone- $\kappa^3 O, N, N'$]-zinc(II), $C_{18}H_{16}N_{10}O_2Zn$

<https://doi.org/10.1515/ncrs-2019-0813>

Received November 4, 2019; accepted December 10, 2019; available online December 27, 2019

Abstract

$C_{18}H_{16}N_{10}O_2Zn$, triclinic, $P\bar{1}$ (no. 2), $a = 10.218(7)$ Å, $b = 10.490(7)$ Å, $c = 11.101(8)$ Å, $\alpha = 79.590(11)^\circ$, $\beta = 65.913(12)^\circ$, $\gamma = 66.785(10)^\circ$, $V = 998.0(12)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0516$, $wR_{ref}(F^2) = 0.1323$, $T = 293(2)$ K.

CCDC no.: 1971033

The crystal 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

Cyanoacetic acid hydrazide (0.099 g, 1 mmol) and 2-aceto-pyrazine (0.122 g, 1 mmol) were dissolved in

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.12 × 0.10 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.27 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
θ_{max} , completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	5099, 3480, 0.030
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2663
$N(param)_{refined}$:	282
Programs:	Bruker programs [1], SHELX [2, 3]

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

Atom	x	y	z	U_{iso}^*/U_{eq}
Zn1	0.26960(5)	0.28380(5)	0.21019(5)	0.0361(2)
O1	0.3652(3)	0.3891(3)	0.0410(3)	0.0405(7)
O2	0.3943(3)	0.3020(3)	0.3153(3)	0.0420(7)
N1	0.0678(4)	0.2763(4)	0.3943(4)	0.0393(9)
N2	−0.2002(5)	0.3050(5)	0.6139(4)	0.0637(13)
N3	0.0973(4)	0.4744(3)	0.2202(3)	0.0358(8)
N4	0.1297(4)	0.5698(3)	0.1181(4)	0.0396(9)
N5	0.3674(7)	0.5082(6)	−0.3012(6)	0.0881(17)
N6	0.2242(4)	0.1516(3)	0.1037(4)	0.0369(9)
N7	0.1988(5)	−0.0471(4)	−0.0124(4)	0.0541(11)
N8	0.4061(3)	0.0815(3)	0.2359(3)	0.0318(8)
N9	0.4924(4)	0.0608(3)	0.3114(4)	0.0377(9)
N10	0.8599(6)	0.0908(6)	0.2995(6)	0.0905(18)
C1	0.0539(5)	0.1734(5)	0.4812(5)	0.0525(13)
H1	0.1355	0.0891	0.4688	0.063*
C2	−0.0808(6)	0.1894(6)	0.5906(5)	0.0619(15)
H2	−0.0865	0.1150	0.6499	0.074*
C3	−0.1861(5)	0.4083(5)	0.5267(5)	0.0500(13)
H3	−0.2685	0.4920	0.5399	0.060*
C4	−0.0527(4)	0.3959(4)	0.4162(4)	0.0357(10)
C5	−0.0319(5)	0.5097(4)	0.3162(4)	0.0368(10)
C6	−0.1548(5)	0.6503(5)	0.3329(5)	0.0555(14)
H6A	−0.1108	0.7172	0.2804	0.083*
H6B	−0.2009	0.6746	0.4241	0.083*
H6C	−0.2316	0.6492	0.3047	0.083*
C7	0.2723(5)	0.5102(4)	0.0337(4)	0.0372(10)
C8	0.3250(5)	0.6015(4)	−0.0838(4)	0.0424(11)
H8A	0.2487	0.6947	−0.0709	0.051*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H8B	0.4202	0.6066	−0.0898	0.051*
C9	0.3484(6)	0.5491(5)	−0.2065(6)	0.0515(13)
C10	0.1357(5)	0.1865(5)	0.0346(5)	0.0459(12)
H10	0.0804	0.2797	0.0244	0.055*
C11	0.1238(6)	0.0869(6)	−0.0229(5)	0.0532(13)
H11	0.0603	0.1156	−0.0708	0.064*
C12	0.2876(5)	−0.0814(5)	0.0588(5)	0.0447(11)
H12	0.3408	−0.1748	0.0706	0.054*
C13	0.3031(4)	0.0153(4)	0.1149(4)	0.0327(9)
C14	0.4030(4)	−0.0227(4)	0.1921(4)	0.0325(9)
C15	0.4917(5)	−0.1708(4)	0.2130(5)	0.0430(11)
H15A	0.5692	−0.1757	0.2434	0.065*
H15B	0.5395	−0.2186	0.1314	0.065*
H15C	0.4237	−0.2134	0.2778	0.065*
C16	0.4757(4)	0.1837(4)	0.3441(4)	0.0350(10)
C17	0.5659(5)	0.1759(5)	0.4275(4)	0.0424(11)
H17A	0.5330	0.2673	0.4609	0.051*
H17B	0.5426	0.1139	0.5027	0.051*
C18	0.7300(6)	0.1276(6)	0.3547(5)	0.0534(13)

methanol (20 mL). The reaction mixture was refluxed for 1 h and cooled to room temperature. Then zinc(II) acetate dihydrate (0.110 g, 0.5 mmol) was added. After stirring for 1 h, the mixture was filtered and set aside to crystallize for several days, giving yellow block crystals.

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [3]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Discussion

Hydrazones derived from 2-aceto-pyrazine and their metal complexes have been widely investigated mainly due to their excellent biological activities [4–6]. However, most research

has focused on aryl-hydrazones and investigations on alkyl-hydrazones are relatively few [4–7]. Herein, the title complex was synthesized and characterized by X-ray diffraction in this work.

In the title crystal structure, the central Zn(II) ion is six-coordinated to two anionic hydrazone ligands by a pair of ON₂ donor sets, thus giving a distorted octahedral coordination geometry. The C–O bond of the hydrazone ligand is in the enolate form, which is supported by the distance of C–O bond being 1.264(5)/1.263(5) Å for C16–O2 and C7–O1, respectively, similar to what was found in other hydrazone ligands [6, 7]. The structure is similar to that of the Zn(II) complex with the 2-aceto-pyrazine benzoylhydrazone ligand [6]. As expected, there exist no classical hydrogen bonds in the crystal.

Acknowledgements: This work is supported by National Natural Science Foundation of China (grant no. 21671124).

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