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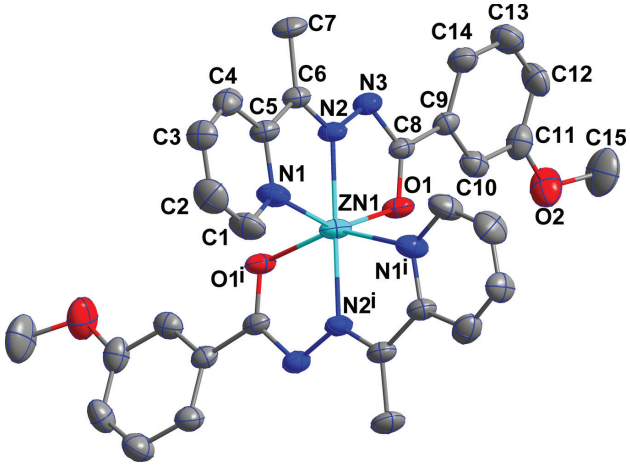
Crystal structure of bis(3-methoxy-*N*-(1-(pyridin-2-yl)ethylidene)benzohydrazonato κ³*O*,*N*,*N*′) zinc(II), $C_{30}H_{28}N_6O_4Zn$



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Abstract

$C_{30}H_{28}N_6O_4Zn$, orthorhombic, *Aba*2 (no. 41), $a = 12.040(9)$ Å, $b = 22.596(18)$ Å, $c = 10.344(10)$ Å, $V = 2814(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0463$, $wR_{ref}(F^2) = 0.1435$, $T = 296(2)$ K.

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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

3-Methoxybenzohydrazonic acid (0.166 g, 1 mmol) and 2-aceto-pyridine systematic name: 1-(pyridin-2-yl)ethan-1-one; (0.121 g, 1 mmol) were dissolved in methanol (20 mL). The reaction mixture was refluxed for 1 h and cooled to room tem-

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Table 1: Data collection and handling.

Crystal:	Yellow plate
Size:	0.15 × 0.12 × 0.06 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.92 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6692, 1968, 0.064
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1235
$N(param)_{refined}$:	188
Programs:	SHELX [1], Bruker [2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.5000	0.5000	0.23455(19)	0.0561(4)
N1	0.4286(6)	0.5587(4)	0.0735(7)	0.063(2)
N2	0.3306(5)	0.4821(3)	0.2291(9)	0.0525(17)
N3	0.2897(5)	0.4393(3)	0.3100(6)	0.0541(18)
O1	0.4779(4)	0.4300(3)	0.3677(6)	0.0596(17)
O2	0.4758(7)	0.2707(3)	0.7010(7)	0.103(3)
C1	0.4782(9)	0.5941(5)	−0.0097(12)	0.082(3)
H1	0.5552	0.5976	−0.0071	0.099*
C2	0.4189(11)	0.6263(5)	−0.1014(10)	0.089(4)
H2	0.4570	0.6515	−0.1568	0.106*
C3	0.3103(9)	0.6220(4)	−0.1117(8)	0.064(3)
H3	0.2719	0.6431	−0.1745	0.077*
C4	0.2570(11)	0.5867(4)	−0.0303(10)	0.072(3)
H4	0.1801	0.5834	−0.0355	0.086*
C5	0.3151(6)	0.5537(4)	0.0648(8)	0.053(2)
C6	0.2623(7)	0.5121(4)	0.1541(8)	0.051(2)
C7	0.1383(7)	0.5031(4)	0.1578(9)	0.070(3)
H7A	0.1222	0.4621	0.1736	0.106*
H7B	0.1067	0.5147	0.0765	0.106*
H7C	0.1069	0.5268	0.2257	0.106*
C8	0.3757(6)	0.4136(4)	0.3750(7)	0.049(2)
C9	0.3439(6)	0.3628(4)	0.4576(7)	0.049(2)
C10	0.4191(7)	0.3407(4)	0.5452(8)	0.061(2)
H10	0.4874	0.3593	0.5560	0.074*
C11	0.3928(9)	0.2900(4)	0.6187(9)	0.071(3)
C12	0.2918(10)	0.2623(4)	0.6046(11)	0.078(3)
H12	0.2738	0.2293	0.6541	0.093*
C13	0.2170(9)	0.2849(5)	0.5142(11)	0.084(3)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H13	0.1489	0.2661	0.5029	0.101*
C14	0.2415(8)	0.3346(4)	0.4407(8)	0.065(3)
H14	0.1905	0.3490	0.3810	0.078*
C15	0.4678(11)	0.2122(5)	0.7544(16)	0.127(5)
H15A	0.4447	0.1851	0.6883	0.190*
H15B	0.4143	0.2121	0.8233	0.190*
H15C	0.5389	0.2004	0.7874	0.190*

perature. Then zinc(II) acetate dihydrate (0.092 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 plate crystals.

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [1]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters. The Flack parameter x = 0.02(3) [1] was determined using 274 quotients [(I+) − (I−)]/[(I+) + (I−)].

Comment

Hydrazones are an important class of ligands with interesting coordination properties due to the presence of several atoms which may coordinate, and are widely applied [3]. In particular, pyridine-containing hydrazones and their metal complexes have been widely investigated mainly due to their excellent biological activities [4–6]. As part of our continuous work, the title complex was synthesized and characterized by X-ray diffraction.

In the title structure, the asymmetric unit contains one half of the complex with Zn1 atom lying on the two fold rotational axis [see the figure, symmetry code: (i) $-x+1$, $-y+1$, z]. The C=O bonds of the hydrazone ligands are enolized, which could be confirmed by the C—O (C8—O1) bond lengths of 1.287(9) Å [4, 5, 7]. The central Zn(II) ion with a distorted octahedral coordination geometry is surrounded by two anionic ligands with N_2O donor set. As expected, there exist no classical hydrogen bonds in the crystal.

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