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Crystal structure of bis[3-methoxy-*N*-(1-(pyrazin-2-yl)ethylidene)benzohydrazonato- $\kappa^3 O, N, N'$] nickel(II), $C_{28}H_{26}N_8O_4Ni$

Table 1: Data collection and handling.

Crystal:	Black block
Size:	0.14 × 0.12 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.78 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12403, 2350, 0.078
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1548
$N(\text{param})_{\text{refined}}$:	188
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.5000	0.10103(8)	0.2500	0.0385(2)
O1	0.4855(2)	0.2411(3)	0.18343(11)	0.0443(7)
O2	0.1264(3)	0.3301(3)	0.00923(13)	0.0678(10)
N1	0.4385(3)	−0.0470(3)	0.30800(13)	0.0430(8)
N2	0.3241(3)	−0.2329(4)	0.37682(16)	0.0640(11)
N3	0.3356(3)	0.0996(3)	0.23360(12)	0.0379(8)
N4	0.2944(2)	0.1812(3)	0.19036(13)	0.0406(8)
C1	0.4941(4)	−0.1253(5)	0.34492(18)	0.0542(11)
H1	0.5734	−0.1188	0.3476	0.065*
C2	0.4372(4)	−0.2155(5)	0.3792(2)	0.0656(14)
H2	0.4792	−0.2669	0.4052	0.079*
C3	0.2690(4)	−0.1549(5)	0.33907(18)	0.0529(11)
H3	0.1901	−0.1642	0.3357	0.063*
C4	0.3235(3)	−0.0612(4)	0.30484(16)	0.0387(9)
C5	0.2656(3)	0.0260(4)	0.26297(15)	0.0383(9)
C6	0.1402(3)	0.0258(5)	0.25562(18)	0.0497(11)
H6A	0.1121	0.1170	0.2575	0.074*
H6B	0.1057	−0.0269	0.2857	0.074*
H6C	0.1211	−0.0128	0.2189	0.074*
C7	0.3822(3)	0.2456(4)	0.16586(16)	0.0393(9)
C8	0.3525(3)	0.3256(4)	0.11449(15)	0.0396(9)
C9	0.2547(3)	0.2971(4)	0.08416(16)	0.0433(10)
H9	0.2067	0.2282	0.0969	0.052*
C10	0.2259(4)	0.3687(4)	0.03504(17)	0.0486(11)
C11	0.2960(4)	0.4720(5)	0.01621(19)	0.0541(12)
H11	0.2772	0.5218	−0.0164	0.065*
C12	0.3942(4)	0.4999(5)	0.04672(18)	0.0538(11)
H12	0.4421	0.5690	0.0341	0.065*
C13	0.4234(4)	0.4292(4)	0.09489(18)	0.0490(11)
H13	0.4904	0.4500	0.1146	0.059*

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Abstract

$C_{28}H_{26}N_8O_4Ni$, orthorhombic, *Pbcn* (no. 60), $a = 11.675(9)$ Å, $b = 9.884(8)$ Å, $c = 23.225(17)$ Å, $V = 2680(4)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0495$, $wR_{\text{ref}}(F^2) = 0.1329$, $T = 296$ 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-Methoxy-benzoic acid hydrazide (0.166 g, 1 mmol) and 2-aceto-pyrazine (0.122 g, 1 mmol) was dissolved in methanol (20 mL). The reaction mixture was refluxed for 1 h and cooled to room temperature. Then nickel(II) acetate tetrahydrate (0.124 g, 0.5 mmol) was added. After stirring for 1 h, the mixture was filtered and set aside to crystallize for several days, giving black blocks.

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C14	0.0965(4)	0.3910(6)	−0.04418(19)	0.0753(16)
H14A	0.1565	0.3755	−0.0717	0.113*
H14B	0.0865	0.4866	−0.0388	0.113*
H14C	0.0265	0.3522	−0.0581	0.113*

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [4]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Comment

Pyrazine-contained hydrazone and their metal complexes have been widely investigated due to their excellent biological activities [4–8]. The title complex was synthesized and characterized by X-ray diffraction in this work.

In the title structure, the asymmetric unit contains a half of the complex with Ni1 atom lying on the two fold rotational axis [Figure 1, symmetry code: (i) $-x + 1, y, -z + 1/2$]. The C=O bonds of the hydrazone ligands are enolized, as confirmed by the C–O (C7–O1) bond lengths of 1.275(4) Å [4, 6, 8]. The central Ni(II) ion with a distorted octahedral coordination geometry is surrounded by two anionic ligands with symmetry related N₂O donor sets. The structure is similar as those of transition metal complexes with 2-aceto-pyrazine

hydrazones [4, 6, 7], and similar crystallographic properties like the methoxy-derivative [8]. As expected, there exist no classical hydrogen bonds in the crystal.

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