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Crystal structure of bis(3-methyl-*N*-(1-(pyrazin-2-yl)ethylidene)benzohydrazonato- $\kappa^3 O, N, N'$) nickel(II), $C_{28}H_{26}N_8O_2Ni$

Table 1: Data collection and handling.

Crystal:	Black block
Size:	0.17 × 0.15 × 0.14 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.81 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} :	6034, 2012, 0.077
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1306
$N(\text{param})_{\text{refined}}$:	179
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.0000	0.5000	0.55930(15)	0.0404(3)
O1	0.0102(4)	0.4242(2)	0.4204(5)	0.0507(14)
N1	0.0663(5)	0.5596(3)	0.7183(7)	0.0518(17)
N2	0.1822(8)	0.6144(3)	0.9289(7)	0.072(2)
N3	0.1597(4)	0.4778(2)	0.5641(8)	0.0439(13)
N4	0.1990(5)	0.4298(3)	0.4844(6)	0.0471(15)
C1	0.0130(8)	0.5941(4)	0.8078(9)	0.068(2)
H1	−0.0645	0.6006	0.7994	0.081*
C2	0.0698(10)	0.6204(4)	0.9124(10)	0.078(3)
H2	0.0290	0.6434	0.9747	0.094*
C3	0.2374(9)	0.5803(3)	0.8388(8)	0.058(2)
H3	0.3155	0.5757	0.8460	0.069*
C4	0.1796(6)	0.5509(3)	0.7324(7)	0.0432(18)
C5	0.2312(6)	0.5062(3)	0.6400(7)	0.0418(17)
C6	0.3546(6)	0.4930(3)	0.6417(7)	0.056(2)
H6A	0.3668	0.4498	0.6625	0.084*
H6B	0.3905	0.5186	0.7079	0.084*
H6C	0.3862	0.5022	0.5557	0.084*
C7	0.1111(7)	0.4044(4)	0.4200(7)	0.0437(19)
C8	0.1393(6)	0.3458(3)	0.3439(8)	0.0424(17)
C9	0.2404(7)	0.3147(3)	0.3705(8)	0.054(2)
H9	0.2911	0.3301	0.4333	0.065*
C10	0.2637(9)	0.2597(4)	0.3006(14)	0.071(2)
H10	0.3310	0.2385	0.3168	0.085*
C11	0.1895(8)	0.2367(4)	0.2091(9)	0.063(2)
H11	0.2075	0.2003	0.1632	0.075*
C12	0.0875(8)	0.2664(4)	0.1829(7)	0.057(2)
C13	0.0641(6)	0.3216(3)	0.2532(7)	0.047(2)
H13	−0.0039	0.3423	0.2380	0.056*
C14	0.0059(8)	0.2374(4)	0.0874(9)	0.084(3)
H14A	−0.0701	0.2441	0.1183	0.125*
H14B	0.0203	0.1934	0.0815	0.125*
H14C	0.0149	0.2560	0.0010	0.125*

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Abstract

$C_{28}H_{26}N_8O_2Ni$, orthorhombic, *Ab*a2 (no. 41), $a = 11.813(10)$ Å, $b = 21.433(17)$ Å, $c = 10.030(8)$ Å, $V = 2539(4)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0457$, $wR_{\text{ref}}(F^2) = 0.0992$, $T = 296(2)$ K.

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The molecular structure is shown in the figure (Hydrogen atoms are omitted for clarity). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

3-Methyl-benzoic acid hydrazide (0.151 g, 1 mmol) and 2-aceto-pyrazine (0.124 g, 1 mmol) were dissolved in methanol

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(20 mL). The reaction mixture was refluxed for 1 h and cooled to room temperature. Then nickel(II) acetate tetrahydrate (0.125 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 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.

Comment

As Schiff bases, hydrazones are an important class of ligands with interesting ligation properties due to the presence of several coordination sites, and are widely applied [4]. In particular, pyrazine-contained hydrazone and their metal complexes have been widely investigated mainly due to their excellent biological activities [5–8]. Herein, the title complex is reported, which was 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 [symmetry code: (i) $-x, -y + 1, z$]. The C=O bond of the hydrazone ligand is enolized, which could be confirmed by the bond length of C–O being 1.265(8) Å [5–7]. All bond distances and angles are in their normal ranges, and can be compared with those in previously reported Co(II) or Ni(II) complexes with pyrazine hydrazones [6–8] as well as with a corresponding Ni(II) complex [9]. The central Ni(II) ion with a distorted octahedral coordination geometry is surrounded by two anionic ligands with the ON₂ donor set for

each ligand, which is the most common coordination mode in pyrazine hydrazono complexes [6–8]. As expected, there exist no classical hydrogen bonds in the crystal.

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