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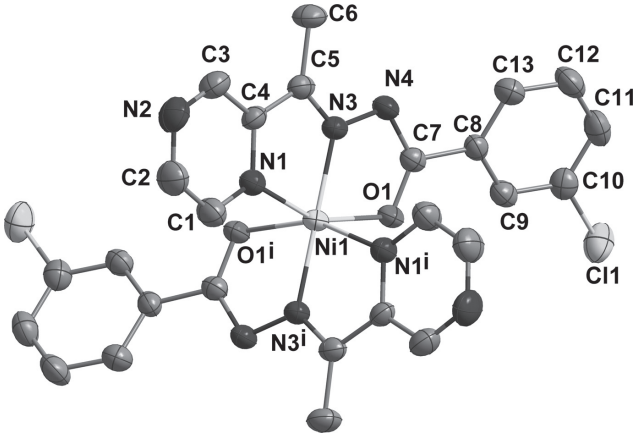
Crystal structure of bis(3-chloro-*N*-(1-(pyrazin-2-yl)ethylidene)benzohydrazonato-k^3*N*,*N*′,*O*) nickel(II), $C_{26}H_{20}N_8O_2Cl_2Ni$



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Abstract

$C_{26}H_{20}N_8O_2Cl_2Ni$, orthorhombic, *Pbcn* (no. 60), $a = 11.737(10)$ Å, $b = 9.309(8)$ Å, $c = 23.72(2)$ Å, $V = 2592(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0361$, $wR_{ref}(F^2) = 0.0934$, $T = 296$ K.

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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

3-Chloro-benzo hydrazide (0.171 g, 1 mmol) and 2-aceto-pyrazine (0.124 g, 1 mmol) were dissolved in methanol (20 mL). The reaction mixture was refluxed for 1 h and cooled to room temperature. Then nickel(II) acetate tetrahydrate

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Table 1: Data collection and handling.

Crystal:	Black block
Size:	0.20 × 0.12 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.00 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11769, 2281, 0.069
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1534
$N(param)_{refined}$:	178
Programs:	Bruker programs [1], SHELX [2, 3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.5000	0.19669(6)	0.2500	0.02913(18)
Cl1	0.48210(8)	−0.25552(11)	0.50479(4)	0.0566(3)
O1	0.51411(16)	0.0532(2)	0.31779(8)	0.0337(5)
N1	0.5600(2)	0.3494(3)	0.19229(10)	0.0345(6)
N2	0.6715(3)	0.5390(3)	0.12105(12)	0.0556(9)
N3	0.6643(2)	0.1996(3)	0.26672(10)	0.0307(6)
N4	0.7039(2)	0.1241(3)	0.31253(10)	0.0345(6)
C1	0.5031(3)	0.4290(4)	0.15527(14)	0.0457(9)
H1	0.4242	0.4213	0.1531	0.055*
C2	0.5594(3)	0.5228(4)	0.12002(14)	0.0559(11)
H2	0.5171	0.5769	0.0945	0.067*
C3	0.7285(3)	0.4582(4)	0.15771(14)	0.0458(9)
H3	0.8074	0.4665	0.1593	0.055*
C4	0.6746(2)	0.3617(3)	0.19372(12)	0.0316(7)
C5	0.7334(3)	0.2742(3)	0.23623(12)	0.0334(8)
C6	0.8595(3)	0.2754(4)	0.24378(14)	0.0461(9)
H6A	0.8781	0.2408	0.2808	0.069*
H6B	0.8874	0.3717	0.2394	0.069*
H6C	0.8941	0.2145	0.2160	0.069*
C7	0.6146(2)	0.0602(3)	0.33724(12)	0.0310(7)
C8	0.6396(3)	−0.0035(3)	0.39356(13)	0.0324(7)
C9	0.5617(3)	−0.0939(3)	0.41903(13)	0.0365(8)
H9	0.4957	−0.1204	0.4000	0.044*
C10	0.5819(3)	−0.1446(4)	0.47267(14)	0.0396(8)
C11	0.6793(3)	−0.1075(4)	0.50163(14)	0.0492(10)
H11	0.6926	−0.1433	0.5376	0.059*
C12	0.7564(3)	−0.0169(4)	0.47649(15)	0.0516(10)
H12	0.8219	0.0100	0.4959	0.062*
C13	0.7374(3)	0.0347(4)	0.42264(13)	0.0422(9)
H13	0.7906	0.0952	0.4059	0.051*

(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. Because of technical problems the accuracy of the lattice parameters is reduced.

Discussion

Hydrazones are a class of ligands with interesting ligation properties, and are widely applied for example as analytical reagents [4]. In particular, pyrazine-contained hydrazone and their metal complexes have been widely investigated mainly due to their excellent biological activities [5–8].

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) $-x+1, y, -z+1/2$]. The C=O bond of the hydrazone ligand is enolized, which could be confirmed by the bond length of C—O being 1.268(3) 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] and especially with a related Cu(II) complex [9]. The central Ni(II) ion with a distorted octahedral coordination geometry is surrounded by two anionic ligands with ON_2 donor set, which is the most common coordination mode in pyrazine hydrazone-metal complexes [6–9]. As expected, there exist no classical hydrogen bonds in the crystal.

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