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Diazidobis(2,2′-biimidazoline)nickel(II)

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.025
wR factor = 0.067
Data-to-parameter ratio = 16.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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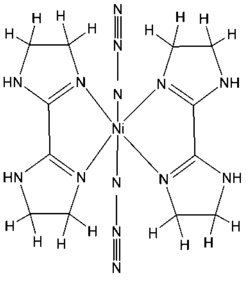
In the crystal structure of the title compound, $[Ni(N_3)_2(C_6H_{10}N_4)_2]$, the Ni^{II} atom, located on an inversion center, adopts an octahedral coordination geometry, with the basal plane formed by four N atoms of two 2,2′-biimidazoline ligands [Ni—N = 2.0701 (13)–2.1099 (14) Å]. The apical positions are occupied by the two N atoms [Ni—N = 2.1851 (14) Å] of the azide anions.

Comment

The bifunctional ligand 2,2′-biimidazoline (abbreviated as biz) is of special interest since, unlike the more 'flat' ligands bi-imidazole, bipyridine and bipyrimidine, it possesses two acidic H atoms at the rear of the molecule. These two NH donor groups can engage in hydrogen bonding to form polymeric aggregates.



(I)

To date, crystal structures of transition metal complexes with biz have been published for Fe^{II}, Fe^{III}, Co^{II} and Co^{III} compounds (Roth *et al.*, 2000; Yoder *et al.*, 2003). More recently, the structures of Cu^{II}, Ni^{II} and Co^{II} complexes with this ligand and the dicyanamide anion have been reported (Mohamadou *et al.*, 2003), as well as the structures of two Cu^{II} compounds (Mohamadou, van Albada, Mills *et al.*, 2004; Mohamadou, van Albada, Driessen *et al.*, 2004). The azide anion used as a ligand in the title complex, (I), is well known for its capability to accept hydrogen bonds.

The Ni^{II} atom is located on an inversion center. The basal plane of the octahedral coordination of the Ni^{II} atom is formed by four N atoms from two bidentate chelating biz ligands at Ni—N distances of 2.0701 (13) and 2.1099 (14) Å. The apical position is occupied by N atoms of two azide groups at an Ni—N distance of 2.1851 (14) Å. The azide anion is nearly linear [N1—N2—N3 = 179.37 (17)°].

The crystal structure is stabilized by intermolecular hydrogen bonding of the uncoordinated NH atoms to N atoms of azide anions with N···N distances of 2.843 (2) and 2.973 (3) Å, creating a two-dimensional array.

In the IR spectrum the azide vibration is observed at 2036 cm^{-1}.

Experimental

The ligand 2,2′-biimidazoline (biz) was prepared according to the method reported in the literature (Wang & Bauman, 1965). The Ni complex was synthesized by mixing biz (2 mmol) and nickel(II) perchlorate hexahydrate (1 mmol) in an ethanol–dimethylformamide (2:1) solution (approximately 40 ml). To the solution, an aqueous solution (3 ml) of sodium azide (1 mmol) was slowly added dropwise and the mixture was left to stand at room temperature. After a few days to one week, green crystals suitable for X-ray diffraction were obtained (yield 72%). Elemental analysis [found (calculated)] for $C_{12}H_{20}NiN_{14}$: C 34.1 (34.4), H 4.4 (4.8), N 46.7% (46.8%).

Crystal data

$[Ni(N_3)_2(C_6H_{10}N_4)_2]$
$M_r = 419.13$
Triclinic, $P\overline{1}$
$a = 7.2960\ (6)$ Å
$b = 7.9420\ (18)$ Å
$c = 8.2080\ (9)$ Å
$\alpha = 97.637\ (14)^\circ$
$\beta = 103.327\ (8)^\circ$
$\gamma = 111.756\ (15)^\circ$
$V = 417.11\ (11)$ Å^3
$Z = 1$
$D_x = 1.669$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6148 reflections
$\theta = 3.2$–27.5°
$\mu = 1.20$ mm^{-1}
$T = 173\ (2)$ K
Block, green
0.30 × 0.30 × 0.28 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.701$, $T_{\max} = 0.716$
6148 measured reflections
1916 independent reflections
1785 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.029$
$\theta_{\max} = 27.5^\circ$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.067$
$S = 1.04$
1916 reflections
120 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0311P)^2 + 0.2437P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.51$ e Å^{-3}
$\Delta\rho_{\min} = -0.58$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N23	2.0701 (13)	Ni1—N1	2.1851 (14)
Ni1—N13$^{\rm i}$	2.1099 (14)		
N23—Ni1—N23$^{\rm i}$	180.0	N23$^{\rm i}$—Ni1—N1	92.19 (5)
N23—Ni1—N13$^{\rm i}$	101.22 (5)	N13$^{\rm i}$—Ni1—N1	85.70 (5)
N23$^{\rm i}$—Ni1—N13$^{\rm i}$	78.78 (5)	N13—Ni1—N1	94.30 (5)
N23—Ni1—N1	87.81 (5)	N3—N2—N1	179.37 (17)

Symmetry code: (i) $-x, -y, -z$.

A crystal was selected for the X-ray measurements and mounted on a glass fiber using the oil-drop method (Kottke & Stalke, 1993) and data were collected at 193 K. H atoms were introduced in calculated positions (C—H = 0.99 Å and N—H = 0.88 Å) and refined with fixed geometry with respect to their carrier atoms [$U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C,N})$].

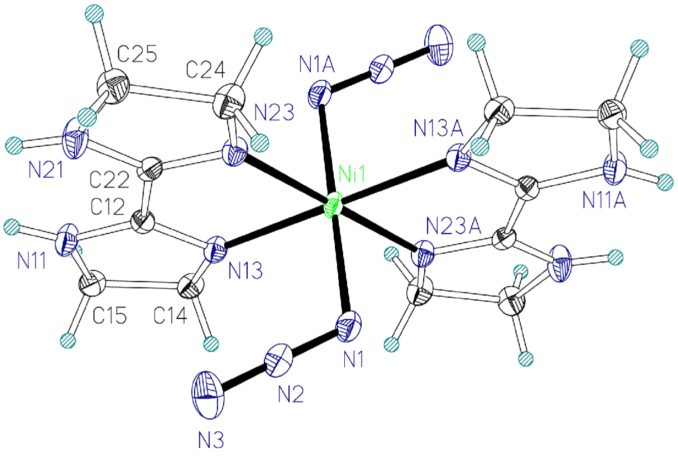


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids. [Symmetry code: (*A*) $-x, -y, z$.]

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *COLLECT*; data reduction: *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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