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Bis(ethylenediamine)nickel(II) dinitrate

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Key indicators

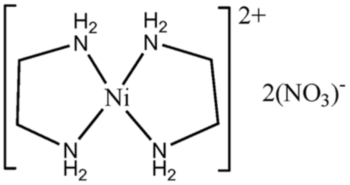
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.091
Data-to-parameter ratio = 13.0

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

In the title complex, $[Ni(C_2H_8N_2)_2](NO_3)_2$, the two ethylenediamine groups, (en), and the nitrate anions are related by an inversion center at the Ni atom. The coordination about the Ni atom can be described as a tetragonally distorted octahedron with four equatorial sites occupied by the N atoms of the (en) groups and the two axial sites by the nitrate anions through a 'semi-coordinating' Ni···O interaction of 2.5539 (15) Å. The remaining O atoms of the nitrate groups participate in N—H···O hydrogen-bonding interactions with the (en) ligand.

Comment

The title complex, (I), was crystallized during ongoing efforts to develop a library of tripodal amine ligands and to study the late first-row transition metal complexes of these ligands (Goldcamp *et al.*, 2003, 2005; Rosa *et al.*, 2001).



(I)

In the structure of (I) (Fig. 1), the two ethylenediamine groups, (en), and the nitrate anions are related by an inversion center at the Ni atom. The type of coordination about the Ni atom is similar to that described by Brown *et al.* (1968) for $[Cu(en)_2](BF_4)_2$, namely a tetragonally distorted octahedron. The four equatorial sites about the Ni atom are occupied by the N atoms of the (en) groups and the two axial sites by the nitrate anions through a 'semi-coordinating' Ni···O interaction. The Ni···O interaction of 2.5539 (15) Å at an angle of 86.92 (8)° relative to the NiN_4 plane is similar to the Cu···O interaction observed for $[Cu(en)_2](ClO_4)_2$ [2.579 (4) Å and 88.0 (1)°; Maxcy & Turnbull, 1999] and $[Cu(en)_2](NO_3)_2$ [2.593 (12) Å and 87 (1)°; Komiyama & Lingafelter, 1964], as well as the Cu···F interaction seen in $[Cu(en)_2](BF_4)_2$ [2.56 (1) Å; Brown *et al.*, 1968].

The Ni—N bond lengths observed in (I) [2.0015 (18) and 2.0171 (18) Å] are shorter than the Ni—N_{en} bonds in $[Ni(en)_2(py)_2](NO_3)_2$ [2.107 (4)–2.145 (3) Å; Baldwin & Krause Bauer, 1999] or $[Ni(en)_3](NO_3)_2$ [2.120 (13) Å; Swink & Atoji, 1960] but compare favorably with the Cu—N_{en} distances (mean 2.020 Å) in $[Cu(NCS)(en)_2]BF_4$ (Koman *et al.*, 1988) and $[Cu(en)_2]X_2$, where X = BF_4, ClO_4, NO_3 and $[(EtO)_2P(S)S]_2$ (Brown *et al.*, 1968; Maxcy & Turnbull, 1999; Komiyama & Lingafelter, 1964; Fun *et al.*, 2002, respectively),

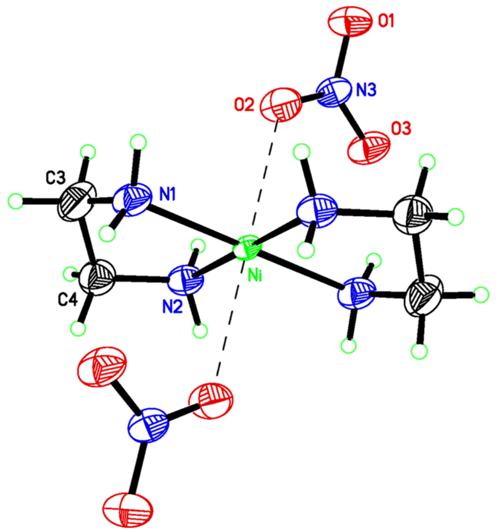


Figure 1
Structure of (I), showing the atomic labeling for the symmetry unique atoms and 50% probability displacement ellipsoids. The 'semi-coordinating' Ni···O interaction is indicated by a dashed line. Unlabeled atoms are related to labeled atoms by $1 - x$, $-y$, $-z$.

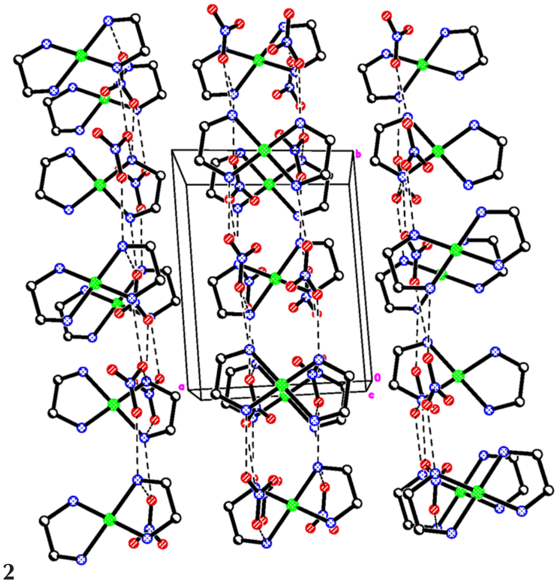


Figure 2
: Packing interactions of (I) in the *ab* plane. N···O interactions are indicated by dashed lines, and H atoms have been omitted for clarity.

as well as the Pt—N$_{en}$ [2.030 (6)–2.043 (7) Å] and Pt$_{en}$ [2.039 (3)–2.046 (3) Å] distances observed by Wiesner & Lingafelter (1966) and Sato *et al.* (1990), respectively. The N—Ni—N angles for (I) (Table 1) are in the 85–95° range, are comparable to those observed in the complexes cited here. The (en) ligand adopts the typical twist conformation, with C3 below and C4 above the NiN$_4$ coordination plane [the respective deviations are −0.283 (4) and 0.391 (4) Å].

While atom O2 of the nitrate anion participates in the 'semi-coordinating' interaction to the Ni atom, atoms O1 and O3 are involved in N—H···O hydrogen-bonding interactions with the (en) ligands. The cations pack in stacked layers, generating a columnar motif in the *ab* plane (Fig. 2), with a 6.355 (1) Å separation between the Ni atoms. The N···O and H···O distances (Table 2), in the range 2.980 (3)–3.008 (2) Å and 2.08–2.26 Å, respectively, are typical and consistent with the complexes discussed.

Experimental

Diffraction quality crystals were obtained by slow evaporation of a methanol solution.

Crystal data

$[Ni(C_2H_8N_2)_2](NO_3)_2$
$M_r = 302.94$
Monoclinic, $P2_1/c$
$a = 8.2382$ (2) Å
$b = 10.0125$ (3) Å
$c = 7.8277$ (2) Å
$\beta = 111.998$ (1)°
$V = 598.66$ (3) Å^3
$Z = 2$

$D_x = 1.681$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 2387 reflections
$\theta = 4.4$–$65.0°$
$\mu = 2.67$ mm^{-1}
$T = 150$ (2) K
Plate, pale purple
$0.29 \times 0.11 \times 0.01$ mm

Data collection

Bruker SMART 6000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003*a*)
$T_{min} = 0.511$, $T_{max} = 0.974$
3389 measured reflections

1027 independent reflections
926 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 67.0°$
$h = -9 \rightarrow 8$
$k = -11 \rightarrow 9$
$l = -9 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.092$
$S = 1.08$
1027 reflections
79 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2 + 0.1767P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—N2	2.0015 (18)	N2—C4	1.474 (3)
Ni—N1	2.0171 (18)	C3—C4	1.510 (3)
N1—C3	1.478 (3)		
N2—Ni—N1	85.08 (7)	C4—N2—Ni	108.47 (14)
N2—Ni—N1^{i}	94.92 (7)	N1—C3—C4	108.30 (19)
C3—N1—Ni	108.60 (14)	N2—C4—C3	108.1 (2)
N2—Ni—N1—C3	−11.64 (15)	Ni—N2—C4—C3	40.4 (2)
N1—Ni—N2—C4	−16.22 (15)	N1—C3—C4—N2	−51.2 (3)
Ni—N1—C3—C4	36.6 (2)		

Symmetry code: (i) $-x + 1, -y, -z$.

Table 2
Hydrogen-bond geometry (Å, °).

	D—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O3^{i}	0.92	2.16	2.980 (3)	148
N1—H1*B*···O3^{ii}	0.92	2.26	3.008 (2)	138
N2—H2*A*···O1^{iii}	0.92	2.09	2.983 (2)	163
N2—H2*B*···O1^{iv}	0.92	2.08	2.998 (2)	178

Symmetry codes: (i) $-x + 1, -y, -z$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x + 1, -y, -z + 1$; (iv) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$.

H atoms were either located or included in calculated positions. H atoms were treated with a riding model (N—H = 0.92 Å and C—H = 0.99 Å), with $U_{iso}(H) = 1.2U_{eq}(N,C)$.

Acta Cryst. (2005). E**61**, m82–m84 Krause Bauer *et al.* • $[Ni(C_2H_8N_2)_2](NO_3)_2$ **m83**

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2003*b*); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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