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Bis{2-[2-(diethylammonio)ethyliminomethyl]-4-nitrophenolato}dimethanolnickel(II) dinitrate

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In the title mononuclear nickel(II) compound, $[Ni(C_{13}H_{19}N_3O_3)_2(CH_4O)_2](NO_3)_2$, the Ni^{II} atom is coordinated by two N and four O atoms, giving an octahedral geometry. The cationic complex lies on a centre of symmetry. In the crystal structure, the molecules are linked through intermolecular O—H···O, N—H···N and N—H···O hydrogen bonds, forming chains running along the *b* axis.

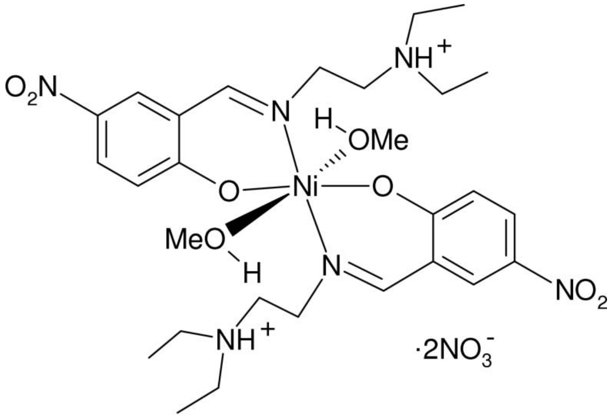
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
Disorder in solvent or counterion
R factor = 0.064
wR factor = 0.180
Data-to-parameter ratio = 15.0

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

Comment

Nickel(II) complexes have received much attention in recent years. As well as being employed as models of metalloenzymes such as [NiFe]-hydrogenases (Marganian *et al.*, 1995), some complexes have been found to have pharmacological and catalytic properties (Brückner *et al.*, 2000; Harrop *et al.*, 2003). As part of our investigations of non-covalent interactions in metal complexes (Chen, 2005), the new title Ni^{II} complex, (I), has been prepared and its crystal structure is presented here.



(I)

The structure of (I) is shown in Fig. 1. The complex consists of an $[Ni(C_{13}H_{19}N_3O_3)_2(CH_3OH)_2]^{2+}$ cation, which possesses a crystallographically imposed centre of symmetry, and two disordered nitrate anions. The coordination sites are occupied by four donor atoms from two Schiff base ligands and two O atoms from two methanol molecules, giving a slightly distorted octahedral geometry. All the bond lengths (Table 1) around the metal centre are comparable with those of similar compounds (Gomes *et al.*, 2000; Arıcı *et al.*, 2005; Jircitano *et al.*, 1990).

In the crystal structure, the molecules are linked through intermolecular O—H···O, N—H···N and N—H···O hydrogen bonds (Table 2), forming chains running along the *b* axis (Fig. 2).

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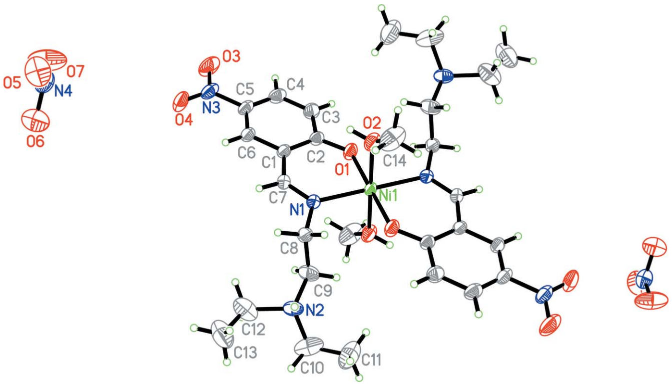

Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. Only the major components of the disordered nitrate anions are shown. Unlabelled atoms are at the symmetry position ($-x$, $2 - y$, $2 - z$).

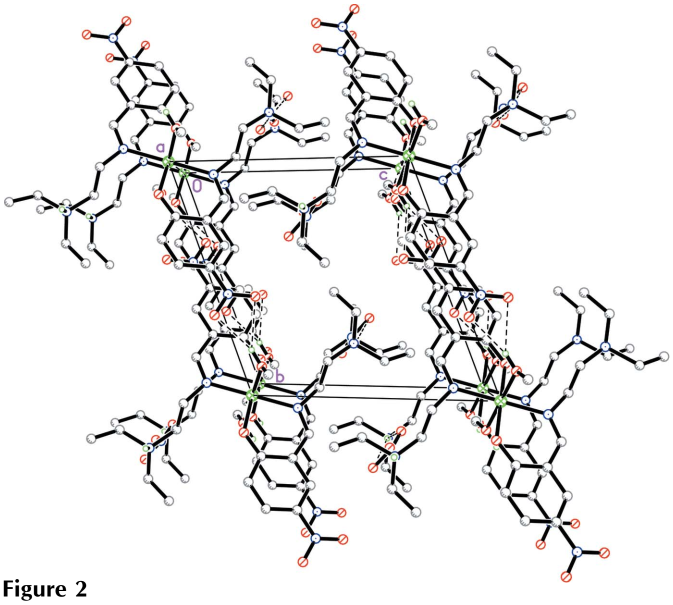

Figure 2
The molecular packing of (I). Intermolecular hydrogen bonds are shown as dashed lines. H atoms and anions have been omitted.

Experimental

All chemicals were of AR grade. 5-Nitro-2-hydroxybenzaldehyde (33.5 mg, 0.2 mmol), *N*,*N*-diethylethane-1,2-diamine (23.2 mg, 0.2 mmol) and $Ni(NO_3)_2 \cdot 6H_2O$ (29.1 mg, 0.1 mmol) were refluxed in methanol (30 ml) for 30 min at 338 K. The mixture was then cooled to room temperature and filtered. After keeping the filtrate in air for 15 d, green block crystals of (I) suitable for X-ray analysis were obtained.

Crystal data

$[Ni(C_{13}H_{19}N_3O_3)_2(CH_4O)_2](NO_3)_2$
$M_r = 777.44$
Triclinic, $P\overline{1}$
$a = 8.351$ (1) Å
$b = 11.000$ (1) Å
$c = 11.097$ (1) Å
$\alpha = 69.092$ (2)°
$\beta = 70.963$ (2)°
$\gamma = 84.048$ (2)°
$V = 900.09$ (16) Å^3

$Z = 1$
$D_x = 1.434$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2165 reflections
$\theta = 2.5$–24.3°
$\mu = 0.62$ mm^{-1}
$T = 298$ (2) K
Block, green
0.12 × 0.11 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\rm min} = 0.930$, $T_{\rm max} = 0.941$
7414 measured reflections

3889 independent reflections
2804 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\rm max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 14$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.180$
$S = 1.00$
3889 reflections
260 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1069P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 1.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.59$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	1.999 (3)	Ni1—N1	2.066 (3)
Ni1—O1$^{\rm i}$	1.999 (3)	Ni1—O2	2.120 (3)
Ni1—N1$^{\rm i}$	2.066 (3)	Ni1—O2$^{\rm i}$	2.120 (3)
O1—Ni1—O1$^{\rm i}$	180	O1$^{\rm i}$—Ni1—O2	91.87 (13)
O1—Ni1—N1$^{\rm i}$	90.79 (11)	N1—Ni1—O2	89.96 (12)
O1—Ni1—N1	89.21 (11)	N1—Ni1—O2$^{\rm i}$	90.04 (12)
N1$^{\rm i}$—Ni1—N1	180	O2—Ni1—O2$^{\rm i}$	180
O1—Ni1—O2	88.13 (13)		

Symmetry code: (i) $-x, -y+2, -z+2$.

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O2—H2⋯N3$^{\rm ii}$	0.85 (4)	2.58 (4)	3.416 (5)	170 (5)
O2—H2⋯O4$^{\rm ii}$	0.85 (4)	2.31 (3)	3.039 (4)	145 (4)
O2—H2⋯O3$^{\rm ii}$	0.85 (4)	2.26 (3)	3.038 (5)	154 (5)
N2—H2*A*⋯N4$^{\rm iii}$	0.90 (3)	2.625 (15)	3.511 (5)	169 (4)
N2—H2*A*⋯O7′$^{\rm iii}$	0.90 (3)	2.55 (3)	3.317 (11)	143 (4)
N2—H2*A*⋯O6$^{\rm iii}$	0.90 (3)	2.53 (3)	3.321 (11)	147 (4)
N2—H2*A*⋯O7$^{\rm iii}$	0.90 (3)	1.96 (2)	2.803 (10)	157 (4)
N2—H2*A*⋯O6′$^{\rm iii}$	0.90 (3)	1.875 (19)	2.749 (9)	164 (5)

Symmetry codes: (ii) $-x, -y+1, -z+2$; (iii) $x, y+1, z$.

Atoms H2 and H2*A* were located in a difference Fourier map and refined isotropically, with the O—H2 distance restrained to 0.84 (1) Å and the N—H2*A* distance restrained to 0.90 (1) Å. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with $U_{\rm iso}({\rm H}) = 1.2$ or $1.5U_{\rm eq}({\rm C})$. The O atoms of the nitrate anions were disordered over two distinct sites with occupancies of 0.536 (2) and 0.464 (2). The N—O and O⋯O distances in both disordered components were restrained to be equal. An unassigned maximum residual density was observed 1.15 Å from atom C12.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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