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[*N*,*N*,*N*′,*N*′-Tetrakis(benzimidazol-2-ylmethyl)-ethane-1,2-diamine]nickel(II) dinitrate methanol solvate

Bing Li, Yu-Hu Wang, Wen Gu and Xin Liu*

Department of Chemistry, Nankai University, Tianjin 300071, People's Republic of China

Correspondence e-mail: liuxin64@nankai.edu.cn

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Key indicators

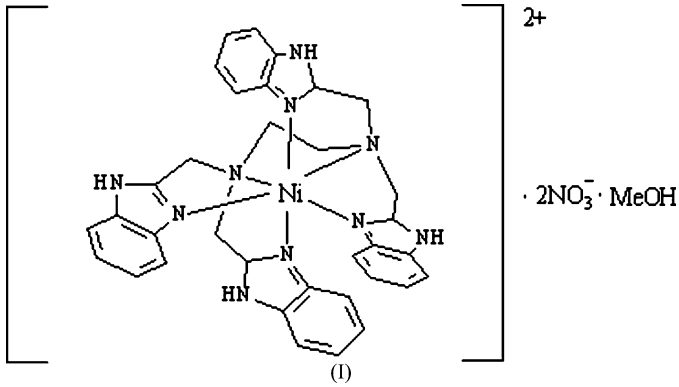
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.044
wR factor = 0.119
Data-to-parameter ratio = 14.8

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

In the title complex, $[Ni(EDTB)](NO_3)_2\cdot CH_3OH$ [EDTB is *N*,*N*,*N*′,*N*′-tetrakis(benzimidazol-2-ylmethyl)ethane-1,2-diamine, $C_{34}H_{32}N_{10}$], the Ni^{II} ion is coordinated by six N atoms of the EDTB ligand to form a distorted octahedral geometry. The crystal packing is stabilized by intermolecular N—H···O, N—H···N and O—H···O hydrogen bonds.

Comment

Some benzimidazole derivatives can be used as building blocks in the synthesis of model complexes (Ogawa *et al.*, 1998; Liu *et al.*, 2002; Blackburn *et al.*, 1989; Plengea *et al.*, 2003). The hexadentate polyfunctional benzimidazole ligand EDTB [EDTB is *N*,*N*,*N*′,*N*′-tetrakis(2-benzimidazolylmethyl)-1,2-ethanediamine] has attracted considerable attention in recent years, and a series of compounds containing this ligand have been reported. Among these, $[Mn(EDTB)(OAc)](OAc)\cdot C_2H_5OH$ (Liao *et al.*, 2001) shows SOD-like activity, while $Cu(EDTB)(NO_3)_2\cdot C_2H_5OH$ (Chen *et al.*, 2004) exhibits catecholase-like activity. As an extension of this work, we have synthesized the title compound, (I), and report its crystal structure here.



(I)

The asymmetric unit of (I) consists of an $[Ni(EDTB)]^{2+}$ cation, two NO_3^- anions and a methanol molecule (Fig. 1). Selected bond lengths and angles are listed in Table 1. The Ni^{II} ion is six-coordinated by four benzimidazole and two amino N atoms, forming a distorted octahedral geometry. The equatorial plane contains the N atoms of two benzimidazole groups and two amino N atoms, while the axial positions are occupied by the N atoms of the other two benzimidazole groups. The Ni atom is displaced by 0.023 (5) Å from the equatorial plane. The crystal packing is stabilized by intermolecular N—H···O, N—H···N and O—H···O hydrogen bonds (Table 2).

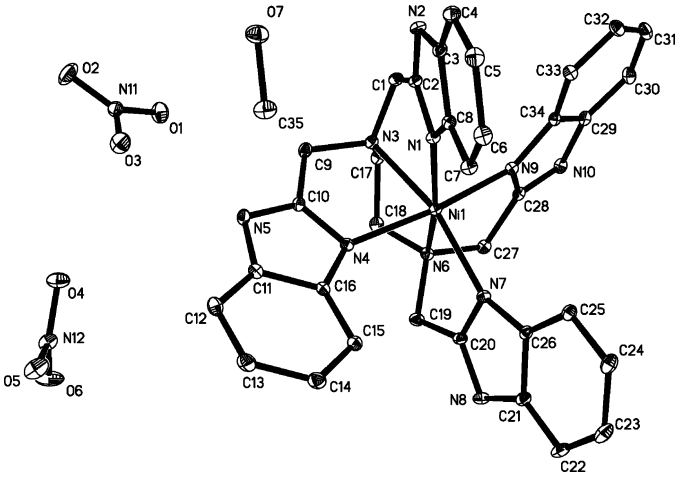


Figure 1
The asymmetric unit of the title compound, with 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

EDTB was prepared as described previously by Gomez-Romero *et al.* (1990). EDTB (1 mmol) was dissolved in hot methanol (15 ml) and a solution of $Ni(NO_3)_2{\cdot}6H_2O$ (1 mmol) in water (10 ml) was added dropwise. The mixture was filtered after continuous stirring for 3 h. Violet single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of the solution at room temperature over a week. Analysis, calculated for $C_{35}H_{36}N_{12}NiO_7$: C 56.55, H 4.34, N 22.61%; found: C 57.46,H 4.52,*N* 23.02%.

Crystal data

$[nI(C_{34}H_{32}N_{10})](NO_3)_2{\cdot}CH_4O$
M_r = 795.47
Triclinic, $P\overline{1}$
a = 11.718 (4) Å
b = 12.103 (4) Å
c = 14.528 (5) Å
α = 91.409 (5)°
β = 103.988 (5)°
γ = 113.291 (5)°
V = 1819.6 (10) Å^3
Z = 2
D_x = 1.452 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 875 reflections
θ = 3.3–25.0°
μ = 0.60 mm^{-1}
T = 293 (2) K
Prism, violet
0.22 × 0.18 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.878, T_{max} = 0.919
10574 measured reflections
7392 independent reflections
5192 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 26.4°
$h = -14 \rightarrow 14$
$k = -15 \rightarrow 14$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.119
S = 1.02
7392 reflections
498 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N1	2.046 (2)	Ni1—N9	2.098 (2)
Ni1—N4	2.094 (2)	Ni1—N6	2.137 (2)
Ni1—N7	2.095 (2)	Ni1—N3	2.186 (2)
N1—Ni1—N4	88.09 (9)	N7—Ni1—N6	78.85 (9)
N1—Ni1—N7	119.12 (9)	N9—Ni1—N6	81.09 (9)
N4—Ni1—N7	92.81 (9)	N1—Ni1—N3	79.18 (9)
N1—Ni1—N9	93.07 (9)	N4—Ni1—N3	80.49 (9)
N4—Ni1—N9	171.40 (8)	N7—Ni1—N3	160.51 (9)
N7—Ni1—N9	94.07 (9)	N9—Ni1—N3	91.35 (8)
N1—Ni1—N6	161.62 (8)	N6—Ni1—N3	83.54 (9)
N4—Ni1—N6	95.20 (9)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O6^{i}	0.86	2.03	2.813 (3)	151
N2—H2···O5^{i}	0.86	2.33	3.035 (3)	140
N2—H2···N12^{i}	0.86	2.53	3.348 (4)	159
N5—H5*A*···O3	0.86	2.05	2.879 (4)	161
N5—H5*A*···O1	0.86	2.48	3.182 (4)	139
N8—H8···O7^{ii}	0.86	1.92	2.763 (3)	168
N10—H10···O3^{iii}	0.86	2.35	3.040 (4)	137
N10—H10···O4^{iii}	0.86	2.37	3.063 (3)	139
O7—H7*A*···O2^{iv}	0.82	2.24	3.032 (5)	164
O7—H7*A*···O1^{iv}	0.82	2.42	3.086 (4)	139

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

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, O—H = 0.82 Å and N—H = 0.86 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-NT* (Bruker, 1998); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-NT* (Bruker, 1998); software used to prepare material for publication: *SHELXTL-NT*.

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