Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diaquadibenzoatobis[2-(4-pyridyl-κ*N*)-4,4,5,5-tetramethylimidazoline-1-oxyl 3-oxide]nickel(II)

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Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.123
Data-to-parameter ratio = 21.4

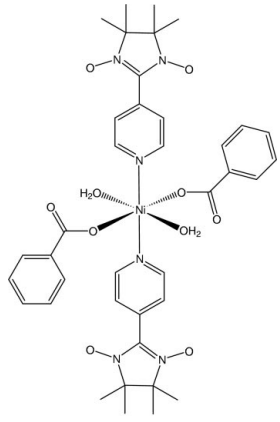
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 14 July 2003
Accepted 18 July 2003
Online 8 August 2003

The Ni atom in $[Ni(O_2CCH_2)_2(PPN)_2(H_2O)_2]$, where PPN is 2-(*p*-pyridyl)-4,4,5,5-tetramethylimidazoline-1-oxyl 3-oxide ($C_{12}H_{16}N_3O_2$), is located on a centre of inversion and exists in a distorted octahedral geometry defined by an N_2O_4 donor set. Complex molecules aggregate into chains mediated by hydrogen-bonding interactions.

Comment

The title compound, (I), is isomorphous with the Mn^{II} species that was investigated for novel magnetic characterisitics owing to the presence of the radical ligand, PPN, *i.e.* 2-(*p*-pyridyl)-4,4,5,5-tetramethylimidazoline-1-oxyl 3-oxide (Fettouhi *et al.*, 1999). The Ni atom is situated on a centre of inversion and exists in a distorted octahedral geometry defined by an all-*trans*-N_2O_4 donor set (Fig. 1 and Table 1).



(I)

The benzoate ligand is effectively mondentate as the Ni···O2 distance is 3.3110 (14) Å. There are both intra- and intermolecular hydrogen-bonding interactions involving the coordinated water molecules and the O2 atoms. The parameters associated with the intramolecular interaction are H···O2 = 1.78 Å, O2···O5 = 2.620 (2) Å and angle at H = 171°. The intermolecular interactions occur between translationally related molecules, along the **a** direction, and are characterized by H···O2^{i} = 2.07 Å, O5···O2^{i} = 2.8468 (19) Å and the angle at H = 154 [symmetry code: (i) 1 + *x*, *y*, *z*]. These interactions lead to the formation of 12-membered [—Ni—O—H···O—C—O—]$_2$ rings and the chains that are thereby formed are separated by regions occupied by the PPN ligands (Fig. 2). Links between the chains are facilitated by C11—H···O4^{ii} contacts, so that H···O4^{ii} = 2.47 Å, C11···O4^{ii} = 3.260 (2) Å and the angle subtended at H = 142° [symmetry

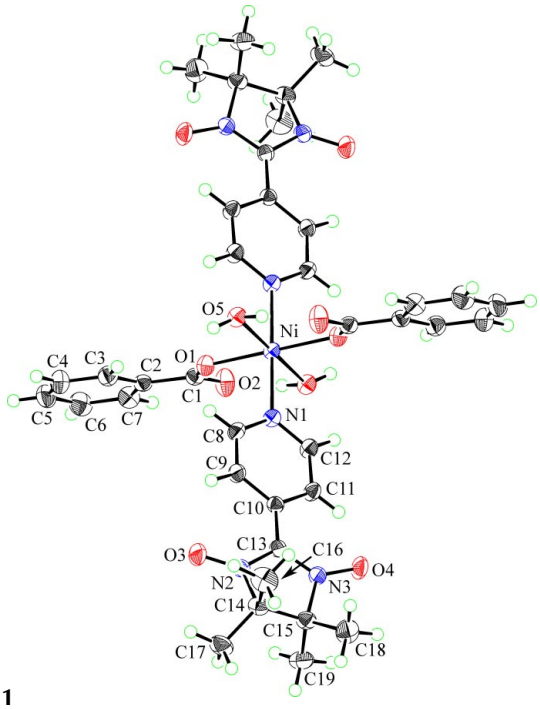


Figure 1
The molecular structure and crystallographic numbering scheme for (I). The unlabelled half of the molecule is generated by the symmetry operation $-x$, $-y$, $-z$. Displacement ellipsoids are drawn at the 50% probability level.

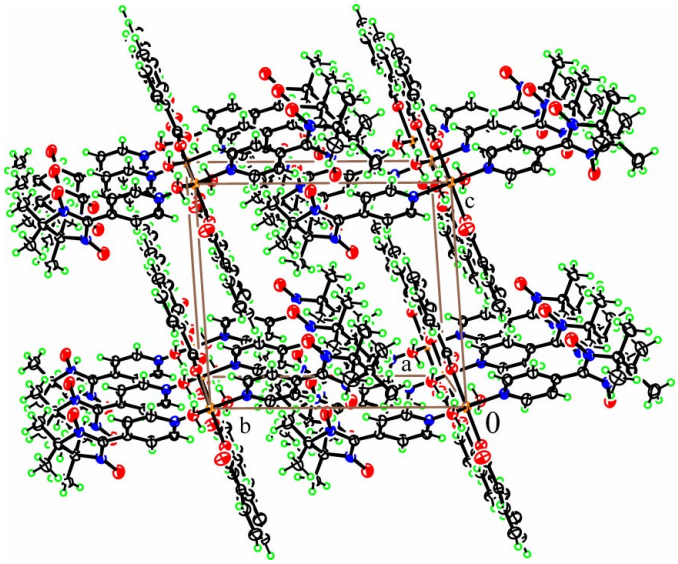


Figure 2
The unit-cell contents for (I) (Farrugia, 1997).

code: (ii) $-x$, $-y$, $1-z$]. Links between the PPN ligands also occur *via* C—H···O interactions so that C17—H···O3^{iii} = 2.55 Å, C17···O3^{iii} = 3.496 (3) Å and the angle at H = 165°, and C16—H···O4^{iv} = 2.54 Å, C16···O4^{iv} = 3.474 (3) Å and the angle at H = 162° [symmetry codes: (iii) $1-x$, $-1-y$, $1-z$; (iv) $1-x$, $-y$, $1-z$].

Experimental

To a solution of $Ni(O_2CC_5H_5)_2{\cdot}4H_2O$ (0.5 mmol) and benzoic acid (1 mmol) in ethanol (25 ml) was added a solid sample of PPN (1 mmol). The solution was stirred for 30 min, after which a small quantity of green solid precipitated. After filtration, the clear solution, kept in the dark, was slowly evaporated at room temperature over a period of one month, yielding green crystals suitable for analysis.

Crystal data

$[Ni(C_7H_5O_2)_2(C_{12}H_{16}N_3O_2)_2(H_2O)_2]$
M_r = 805.52
Triclinic, $P\overline{1}$
a = 6.7001 (3) Å
b = 11.3354 (6) Å
c = 12.8740 (6) Å
α = 86.319 (2)°
β = 87.290 (2)°
γ = 79.486 (2)°
V = 958.73 (8) Å^3
Z = 1
D_x = 1.395 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2446 reflections
θ = 2.4–28.9°
μ = 0.57 mm^{-1}
T = 223 (2) K
Irregular block, green
0.28 × 0.14 × 0.10 mm

Data collection

Bruker AXS SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000; Blessing, 1995)
T_{min} = 0.725, T_{max} = 0.944
8055 measured reflections
5468 independent reflections
4462 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 30.0°
$h = -8 \rightarrow 9$
$k = -15 \rightarrow 14$
$l = -16 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.123
S = 1.05
5468 reflections
256 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—O1	2.0497 (13)	O2—C1	1.256 (2)
Ni—O5	2.1036 (13)	O3—N2	1.272 (2)
Ni—N1	2.1011 (15)	O4—N3	1.274 (2)
O1—C1	1.260 (2)		
O1—Ni—O5	88.52 (5)	O1—Ni—N1^{i}	90.01 (5)
O1—Ni—N1	89.99 (5)	O5—Ni—N1	93.10 (5)
O1—Ni—O5^{i}	91.48 (5)	O5—Ni—N1^{i}	86.90 (5)

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

The carbon-bound H atoms were included in the riding-model approximation, with C—H distances of 0.94 Å (0.97 Å for methyl), U_{iso}(phenyl–H) = 1.2U_{eq}(C) and U_{iso}(methyl–H) = 1.5U_{eq}(C). The water H atoms were located from a difference map and refined, subject to O—H 0.85 (1) Å, H···H 1.39 (1) Å and U_{iso}(H) = 1.5U_{eq}(O).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* and *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *PATTY* in *DIRDIF*92 (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXTL*.

This work is supported by the National Natural Science Foundation of China (grant No. 29961002), the National Natural Science Foundation of Yunnan Province (grant No.

2002FXX05), the National Natural Science Foundation of Yunnan University (grant No. 2002Z001GC), the National Natural Science Foundation of the Educational Committee of Yunnan Province (grant No. 0111158) and the National University of Singapore (R-143-000-139-112).

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