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cyclo-Tetrakis{μ-1-[2-(2-oxidoethylamino)ethyliminomethyl]naphthalen-2-olato}tetranickel(II)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.123
Data-to-parameter ratio = 15.9

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

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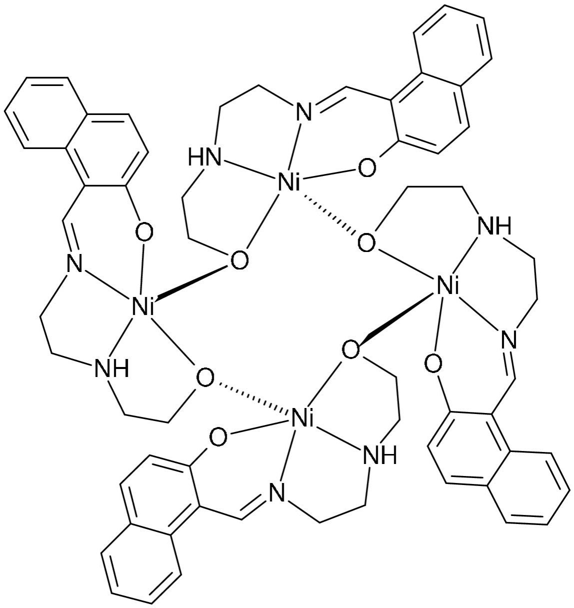
The title tetranuclear nickel(II) compound, $[Ni_4(C_{15}H_{16}N_2O_2)_4]$, is isostructural with the zinc(II) complex reported recently [You, Ma, Zhu & Liu (2004). *Acta Cryst.* E**60**, m1599-m1601]. The complex has $\overline{4}$ crystallographic symmetry. Each Ni^{II} atom has a distorted square-pyramidal geometry. In the basal plane, the Ni atom is coordinated by two N atoms and two O atoms of a Schiff base. The apical position is occupied by another O atom of another Schiff base.

Comment

Recently, we have reported some polynuclear Schiff base complexes (You, Zhu & Liu, 2004*a*; You, Zhu & Liu, 2004*b*; You & Zhu, 2004). As an extension of our work on polynuclear Schiff base complexes, the title tetranuclear nickel(II) compound, (I), is reported here.



(I)

Compound (I) is isostructural with the tetranuclear zinc(II) complex tetrakis{μ-1-[2-(2-oxidoethylamino)ethyliminomethyl]-2-naphtholato}tetrazinc, (II), which we have reported recently (You, Ma *et al.*, 2004). The tetramer complex, (I), has $\overline{4}$ crystallographic symmetry. Each Ni atom has a square-pyramidal geometry, coordinated by two N and three O atoms of two Schiff bases. In (I), all bond lengths and angles (Table 1) are comparable with those in (II).

In the crystal structure of (I), there are no short contacts between the molecules (Fig. 2).

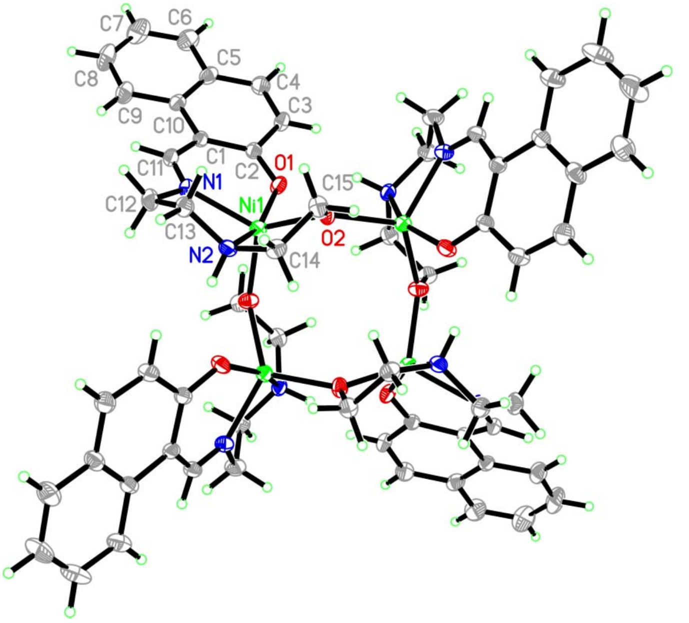


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Unlabelled atoms are related to labelled atoms by the symmetry operators ($\frac{3}{2} - y, x, \frac{1}{2} - z$), ($\frac{3}{2} - x, \frac{3}{2} - y, z$) and ($y, \frac{3}{2} - x, \frac{1}{2} - z$).

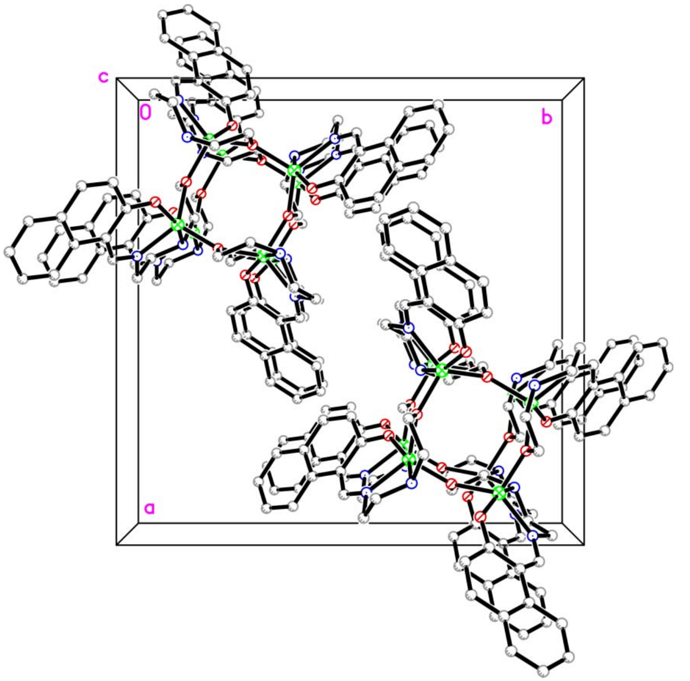


Figure 2
The crystal packing of (I), viewed along the *c* axis. H atoms have been omitted.

Experimental

N-(2-Hydroxyethyl)ethylenediamine (0.2 mmol, 20.8 mg) and salicylaldehyde (0.2 mmol, 24.4 mg) were dissolved in methanol (5 ml). The mixture was stirred for 10 min at room temperature to give a clear yellow solution. To this solution was added nickel oxide powder (0.2 mmol, 15.0 mg), and the mixture was stirred for a further 10 min. The mixture was then transferred to a stainless steel bomb, which was sealed, heated at 423 K for 48 h and cooled gradually to room temperature. Green block-shaped crystals of (I) were formed.

Crystal data

[$Ni_4(C_{15}H_{16}N_2O_2)_4$]
$M_r = 1260.03$
Tetragonal, $P4_2/n$
$a = 18.1814$ (8) Å
$c = 8.5460$ (7) Å
$V = 2825.0$ (3) Å^3
$Z = 2$
$D_x = 1.481$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 1898 reflections
$\theta = 2.1$–$22.4°$
$\mu = 1.38$ mm^{-1}
$T = 298$ (2) K
Block, green
0.34 × 0.28 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.652$, $T_{max} = 0.752$
12894 measured reflections

2917 independent reflections
2333 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 26.5°$
$h = -22 \rightarrow 19$
$k = -22 \rightarrow 22$
$l = -7 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.123$
$S = 1.06$
2917 reflections
184 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2 + 1.4827P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O2^{i}	1.984 (2)	Ni1—N1	2.050 (3)
Ni1—O2	2.009 (2)	Ni1—N2	2.174 (3)
Ni1—O1	2.020 (2)		
O2^{i}—Ni1—O2	116.62 (12)	O1—Ni1—N1	86.42 (9)
O2^{i}—Ni1—O1	105.45 (9)	O2^{i}—Ni1—N2	94.45 (9)
O2—Ni1—O1	95.07 (9)	O2—Ni1—N2	81.65 (9)
O2^{i}—Ni1—N1	109.87 (10)	O1—Ni1—N2	158.99 (9)
O2—Ni1—N1	131.08 (10)	N1—Ni1—N2	80.41 (10)

Symmetry code: (i) $-y + \frac{3}{2}, x, -z + \frac{1}{2}$.

Atom H2 was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. All H atoms attached to C atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$.

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

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