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# (1,8-Di-*n*-propyl-1,3,6,8,10,13-hexaazacyclotetradecane)nickel(II) diperchlorate

**Yan-Wu Li,[a] Hua Xiang,[a] Tong-Bu Lu[a] and Seik Weng Ng[b]***

[a]Instrument Analysis and Research Center, Sun Yat-Sen University, Guangzhou 510275, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

**Key indicators**

Single-crystal X-ray study
*T* = 298 K
Mean σ(C–C) = 0.006 Å
Disorder in solvent or counterion
*R* factor = 0.044
*wR* factor = 0.131
Data-to-parameter ratio = 12.8

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

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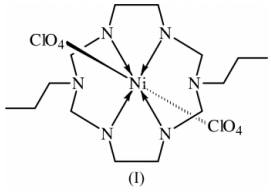
Nickel complexes of 1,8-diorganyl-1,3,6,8,10,13-hexaazacyclotetradecane. Part II.

In the title complex, $[Ni(C_{14}H_{34}N_6)](ClO_4)_2$, the 14-membered hexaazacyclotetradecane macrocycle ring chelates to the Ni atom through its four secondary N atoms. The O atoms of the perchlorate ions are 2.86 (1) and 3.070 (4) Å from the Ni atom, whose geometry is best described as intermediate between octahedral and square pyramidal.

## Comment

This 14-membered hexaazamacrocyclic complex of nickel perchlorate, (I), has an *n*-propyl group in place of a benzyl group (Li *et al.*, 2004). One of the perchlorate ions is disordered. The ordered perchlorate ion has an O atom 3.074 (4) Å from Ni1, while the other perchlorate ion is disordered over two positions, with the O atom of the minor component 2.86 (1) Å from Ni1. The O atom of the ordered perchlorate ion is not considered as being in the coordination sphere. The coordination geometry of the Ni atom is better regarded as intermediate between octahedral and square pyramidal (Fig. 1). Weak hydrogen bonds (Table 2) link the molecules into a linear chain structure (Fig. 2).




(I)

## Experimental

The title compound was synthesized according to the method of Li *et al.* (2004), with *n*-propylamine (8.3 g, 0.15 mmol) in place of benzylamine, in 25% yield. CHN analysis for $C_{14}H_{34}Cl_2N_6O_8Ni$ found: C 30.63, H 6.71, N 15.38%; calculated: C 30.90, H 6.30, N 15.45%.

### *Crystal data*

$[Ni(C_{14}H_{34}N_6)](ClO_4)_2$
$M_r$ = 544.08
Triclinic, $P\overline{1}$
*a* = 8.1634 (6) Å
*b* = 12.411 (1) Å
*c* = 12.518 (1) Å
α = 77.094 (1)°
β = 83.348 (1)°
γ = 71.882 (1)°
*V* = 1173.5 (2) Å$^3$
*Z* = 2
$D_x$ = 1.540 Mg m$^{-3}$
Mo *Kα* radiation
Cell parameters from 2807 reflections
θ = 4.2–29.5°
μ = 1.11 mm$^{-1}$
*T* = 298 (2) K
Plate, yellow
0.42 × 0.25 × 0.08 mm

### *Data collection*

Bruker SMART 1K area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.654, $T_{max}$ = 0.917
6468 measured reflections
3973 independent reflections
3155 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.015
$\theta_{max}$ = 25.0°
*h* = −9 → 9
*k* = −14 → 14
*l* = −14 → 14

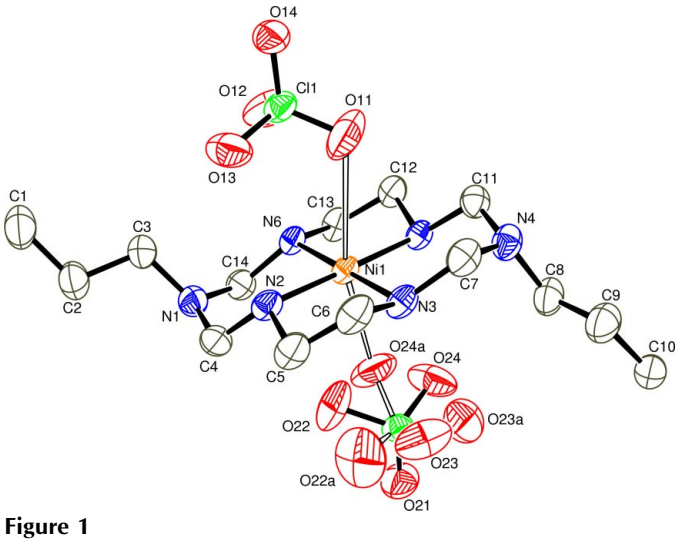



**Figure 1**
*ORTEP*III (Burnett & Johnson, 1996) plot of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) $1-x, 1-y, 1-z$.]

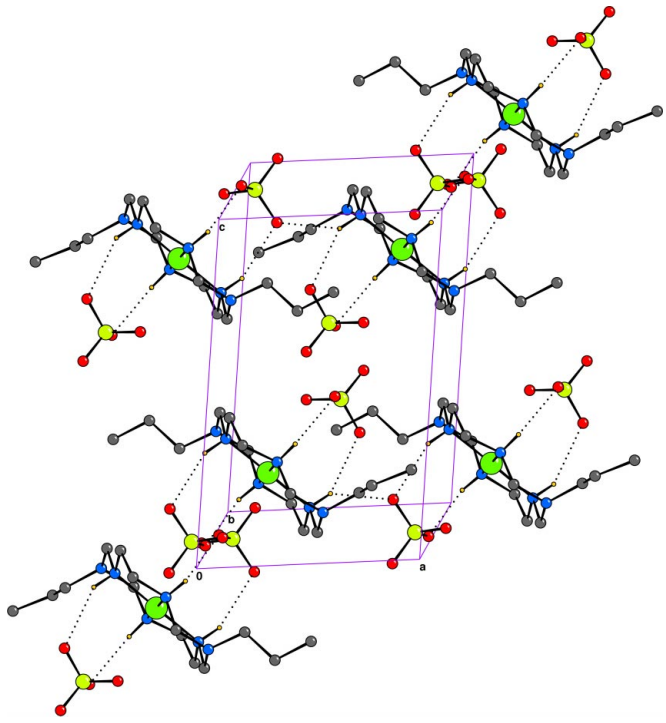



**Figure 2**
*CAMERON* (Watkin *et al.*, 1993) view showing the N—H···F hydrogen-bond interactions within the unit cell. For clarity, the second components of the disordered O atoms have been omitted and only the H atoms attached to nitrogen are shown.

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0832P)^2 + 0.0059P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.044$ | $(\Delta/\sigma)_{max} = 0.004$ |
| $wR(F^2) = 0.131$ | $\Delta\rho_{max} = 0.53$ e Å$^{-3}$ |
| $S = 1.08$ | $\Delta\rho_{min} = -0.33$ e Å$^{-3}$ |
| 3973 reflections | |
| 311 parameters | |
| H-atom parameters constrained | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Ni1—N2 | 1.932 (3) | Ni1—O24*A* | 2.863 (11) |
| Ni1—N6 | 1.934 (2) | Ni1—O11 | 3.060 (4) |
| Ni1—N5 | 1.934 (2) | Ni1—O22 | 3.134 (9) |
| Ni1—N3 | 1.937 (2) | | |
| N2—Ni1—N6 | 92.47 (10) | N2—Ni1—O11 | 92.56 (12) |
| N2—Ni1—N5 | 178.99 (9) | N6—Ni1—O11 | 97.56 (10) |
| N6—Ni1—N5 | 87.05 (9) | N5—Ni1—O11 | 86.62 (11) |
| N2—Ni1—N3 | 87.44 (11) | N3—Ni1—O11 | 82.26 (11) |
| N6—Ni1—N3 | 179.79 (10) | O24*A*—Ni1—O11 | 164.6 (3) |
| N5—Ni1—N3 | 93.03 (11) | N2—Ni1—O22 | 80.5 (2) |
| N2—Ni1—O24*A* | 102.8 (3) | N6—Ni1—O22 | 84.93 (19) |
| N6—Ni1—O24*A* | 82.0 (2) | N5—Ni1—O22 | 100.3 (2) |
| N5—Ni1—O24*A* | 78.0 (3) | N3—Ni1—O22 | 95.2 (2) |
| N3—Ni1—O24*A* | 98.2 (2) | O11—Ni1—O22 | 172.8 (2) |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···O13 | 0.91 | 2.36 | 3.217 (4) | 156 |
| N2—H2···O24$^{i}$ | 0.91 | 2.63 | 3.336 (6) | 135 |
| N3—H3···O23 | 0.91 | 2.40 | 3.250 (7) | 155 |
| N5—H5···O24 | 0.91 | 2.46 | 3.276 (6) | 150 |
| N5—H5···O13$^{ii}$ | 0.91 | 2.52 | 3.258 (4) | 139 |
| N6—H6···O12 | 0.91 | 2.22 | 3.050 (4) | 151 |

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

The nitrogen- and carbon-bound H atoms were placed in calculated positions and were refined in the riding-model approximation (N—H = 0.91 Å, methylene C—H = 0.97 Å and methyl C—H = 0.96 Å]. The displacement parameters were set to 1.2 times $U_{eq}$ of the parent atoms, except for the methyl H atoms, for which the displacement parameters were set at $1.5U_{eq}$(C). Three of the O atoms of one of the perchlorate ions are disordered over two positions by rotation around the Cl—O(non-disordered) axis. The occupancies of the O atoms refined to an approximate 2:1 ratio. The Cl—O distances and O···O interactions in the disordered ion were restrained to reasonable values. There is a short inter-ion contact (O3···O7) of 2.76 Å that is probably an artifact of the disorder.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: ORTEPIII (Burnett & Johnson, 1996) and *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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