

catena-Poly[[*(3,10-diethyl-1,3,5,8,10,12-hexaazacyclotetradecane)nickel(II)- μ -terephthalato*]]

Yan-Wu Li,^a Hua Xiang,^a
Tong-Bu Lu^a and Seik Weng Ng^{b*}

^aInstrument Analysis and Research Center, Sun Yat-Sen University, Guangzhou 510275, People's Republic of China, and ^bDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

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

Key indicators

Single-crystal X-ray study

$T = 298\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

R factor = 0.030

wR factor = 0.081

Data-to-parameter ratio = 17.5

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

In the title complex, $[\text{Ni}(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_{12}\text{H}_{30}\text{N}_6)]_n$, the 14-membered hexaazacyclotetradecane macrocycle ring chelates to the Ni atom through its four secondary N atoms; the Ni atom and the terephthalate anion lie on inversion centers. The terephthalate dianion links adjacent nickel–macrocycle cations through the carboxyl O atoms [$\text{Ni}-\text{O}$ 2.144 (2) Å] into a linear chain.

Received 13 January 2004

Accepted 30 January 2004

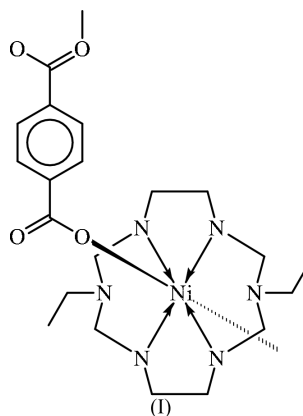
Online 20 February 2004

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

Comment

Among the nickel complexes of the 14-membered hexaazacyclotetradecane macrocycle, the carboxylate derivatives are capable of forming strong Ni–O(carboxylate) bonds (Li *et al.*, 2004). The terephthalate dianion has also been used to bind to Ni in the macrocyclic complex having the $-\text{CH}_2\text{CH}_2\text{OH}$ pendent arm. In the title complex, (I), the two carboxyl $-\text{CO}_2$ groups are twisted with respect to the aromatic ring so as to bond to the Ni atom, but the twist apparently weakens the Ni–O bond [2.129 (5) Å] somewhat [dihedral angles = 12.01 (1) and 17.9 (1)°] (Choi & Suh, 1999). The complex is a rare example of the use of a macrocycle-metal entity in the construction of network structures.

The Ni atom and the terephthalate anion lie on inversion centers. The title compound (Fig. 1) adopts a linear chain structure and the chains are all parallel (Fig. 2). This motif contrasts with that of the $-\text{CH}_2\text{CH}_2\text{OH}$ analog, in which the chains in one layer run approximately perpendicular to the those of the next layer to furnish a plywood-like network arrangement.



Experimental

The title compound was synthesized from ethylamine (6.8 g, 0.15 g) and the sodium salt of terephthalic acid according to the method of Suh *et al.* (1994). CHN analysis for $\text{C}_{20}\text{H}_{34}\text{N}_6\text{NiO}_4$ found: C 49.96, H 7.39, N 17.26%; calculated: C 49.91, H 7.12, N 17.47%.

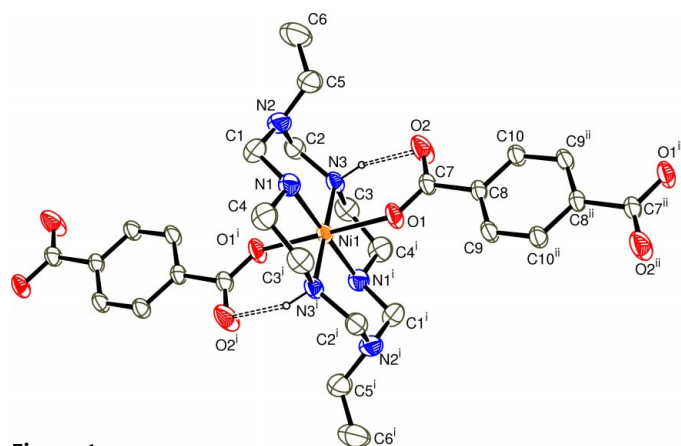


Figure 1
ORTEP (Burnett & Johnson, 1996) plot of a fragment of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. Hydrogen bonds are shown dashed. [Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $2 - x, 1 - y, 2 - z$.]

Crystal data

$[\text{Ni}(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_{12}\text{H}_{30}\text{N}_6)]$	$Z = 1$
$M_r = 481.24$	$D_x = 1.421 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.5818 (5) \text{ \AA}$	Cell parameters from 2995 reflections
$b = 8.3976 (5) \text{ \AA}$	$\theta = 4.0\text{--}30.0^\circ$
$c = 9.7596 (6) \text{ \AA}$	$\mu = 0.90 \text{ mm}^{-1}$
$\alpha = 105.506 (1)^\circ$	$T = 298 (2) \text{ K}$
$\beta = 97.283 (1)^\circ$	Plate, yellow
$\gamma = 105.853 (1)^\circ$	$0.27 \times 0.23 \times 0.08 \text{ mm}$
$V = 562.45 (6) \text{ \AA}^3$	

Data collection

Bruker SMART 1K area-detector diffractometer	2500 independent reflections
φ and ω scans	2397 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.010$
$T_{\text{min}} = 0.792, T_{\text{max}} = 0.931$	$\theta_{\text{max}} = 27.5^\circ$
3389 measured reflections	$h = -9 \rightarrow 9$
	$k = -7 \rightarrow 10$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 0.1456P]$
$R[F^2 > 2\sigma(F^2)] = 0.030$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.081$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.42 \text{ e \AA}^{-3}$
2500 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
143 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters ($\text{\AA}, ^\circ$).

Ni1—N3	2.0574 (13)	Ni1—O1	2.1440 (11)
Ni1—N1	2.0617 (14)		
N3—Ni1—N1	94.25 (6)	N3 ⁱ —Ni1—O1	87.73 (5)
N3 ⁱ —Ni1—N1	85.75 (6)	N1—Ni1—O1	88.64 (6)
N3—Ni1—O1	92.27 (5)	N1 ⁱ —Ni1—O1	91.36 (6)

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

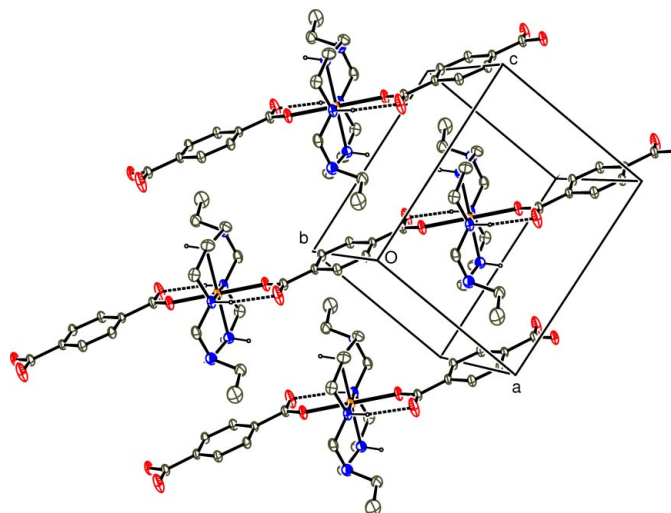


Figure 2
ORTEP (Burnett & Johnson, 1996) plot of the polymeric chains in the structure of (I).

Table 2

Hydrogen-bonding geometry ($\text{\AA}, ^\circ$).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
$\text{N3--H3}\cdots\text{O2}$	0.91	2.03	2.878 (2)	154

The nitrogen- and carbon-bound H atoms were placed at calculated positions and were refined in the riding-model approximation ($\text{N--H} = 0.91 \text{ \AA}$, methylene $\text{C--H} = 0.97 \text{ \AA}$ and phenyl $\text{C--H} = 0.93 \text{ \AA}$), with $U(\text{H}) = 1.2U_{\text{eq}}$ of the parent atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank the National Science Foundation of China (No. 20371051), the Ministry of Education of China and the University of Malaya for supporting this work.

References

- Bruker (2001). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Choi, H. J. & Suh, M. P. (1999). *Inorg. Chem.* **38**, 6309–6312.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Li, Y.-B., Xiang, H., Lu, T.-B. & Ng, S. W. (2004). *Acta Cryst.* **E60**, m309–m311.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Suh, M. P., Shim, B. Y. & Yoon, T.-S. (1994). *Inorg. Chem.* **33**, 5509–5514.