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**Key indicators**

Single-crystal X-ray study  
 T = 293 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$   
 R factor = 0.045  
 wR factor = 0.126  
 Data-to-parameter ratio = 13.8

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

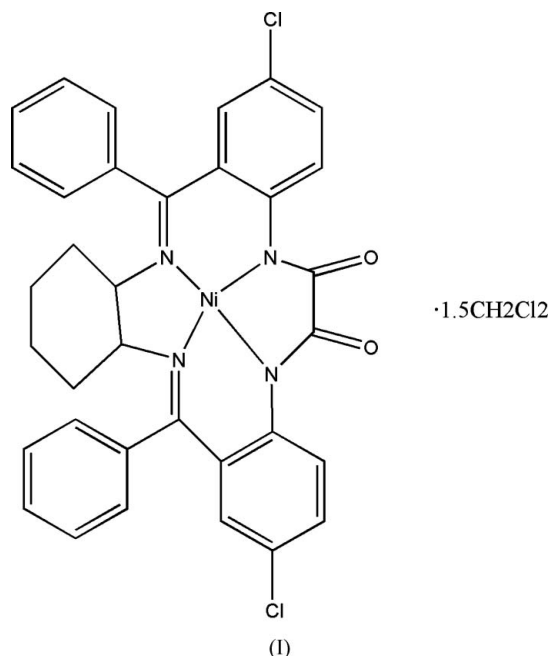
**A new nickel(II) complex  $[\text{NiL}]\cdot 1.5\text{CH}_2\text{Cl}_2$ ,  
 where  $\text{H}_2\text{L}$  is 14,25-dichloro-2,11-diphenyl-  
 tetracyclo[20.4.0.0<sup>4,9</sup>.0<sup>12,17</sup>]hexacos-  
 2,10,12,14,16,22,24,26-octaene-19,20-dione**

In the new nickel(II) complex [14,25-dichloro-2,11-diphenyl-tetracyclo[20.4.0.0<sup>4,9</sup>.0<sup>12,17</sup>]hexacos-2,10,12,14,16,22,24,26-octaene-19,20-dionato(2-)]nickel(II) dichloromethane sesquisolvate,  $[\text{Ni}(\text{C}_{34}\text{H}_{26}\text{Cl}_2\text{N}_4\text{O}_2)]\cdot 1.5\text{CH}_2\text{Cl}_2$ , the nickel(II) ion is four-coordinate and displays approximately square-planar coordination geometry. It binds to two deprotonated oxamide N atoms and two imine N atoms of the macrocyclic ligand. The C atom of one of the two dichloromethane solvent molecules lies on a twofold axis.

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**Comment**

Metal complexes of macrocyclic ligands have been of great interest in coordination chemistry because of their special structures, properties and/or functionalities (Chen & Suslick, 1993; Vigato *et al.*, 1990). It has also been shown that the oxamide group provides an efficient pathway for magnetic exchange between neighbouring metal ions (Wang *et al.*, 2004; Zhang *et al.*, 2003). The type and magnitude of the magnetic coupling will depend on the metal ions used and their coordination environments. (Kahn, 1993).



We report here a new nickel(II) complex,  $[\text{NiL}]\cdot 1.5(\text{CH}_2\text{Cl}_2)$  ( $\text{H}_2\text{L}$  is 14,25-dichloro-2,11-diphenyl-tetracyclo[20.4.0.0<sup>4,9</sup>.0<sup>12,17</sup>]hexacos-2,10,12,14,16,22,24,26-octaene-19,20-dione), in which the macrocyclic ligand coordinates to the nickel atom *via* the two deprotonated oxamide N atoms and the two imine N atoms in an approximately square-planar

coordination geometry (Table 1). The Ni atom resides in the mean plane of the four donor atoms, and their deviations from the mean plane are 0.0887 (14) Å (N1), -0.0922 (15) Å (N2), 0.0910 (15) Å (N3) and -0.0876 (14) Å (N4); atom Ni1 lies -0.0042 (15) Å from this plane. The crystal structure is stabilized by weak C—H...O and C—H...Cl hydrogen bonds (Table 1). A packing diagram is shown in Fig. 2.

## Experimental

All chemicals were of reagent grade and obtained commercially without further purification. The compound 2,2'-(oxalyldiimino)-bis(chlorobenzaldehyde), *L'*, was prepared as described previously (Zhang *et al.*, 2005). A mixture of *L'* (5.73 mmol), 1,2-diaminocyclohexane (17.18 mmol) and Ni(OAc)<sub>2</sub>·4H<sub>2</sub>O (5.73 mmol) was refluxed for 10 h in methanol (50 ml), in the presence of 2M NaOH (2.80 ml). After the mixture had been cooled and filtered, the resulting red polycrystalline powder was dissolved in CH<sub>2</sub>Cl<sub>2</sub> and kept at room temperature for two days to give red crystals suitable for X-ray analysis.

### Crystal data

[Ni(C <sub>34</sub> H <sub>26</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>2</sub> )]·1.5CH <sub>2</sub> Cl <sub>2</sub>	$D_x = 1.542 \text{ Mg m}^{-3}$
$M_r = 779.59$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 985 reflections
$a = 26.745 (7) \text{ \AA}$	$\theta = 3.2\text{--}25.4^\circ$
$b = 12.520 (3) \text{ \AA}$	$\mu = 1.02 \text{ mm}^{-1}$
$c = 22.820 (6) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 118.454 (4)^\circ$	Block, red
$V = 6718 (3) \text{ \AA}^3$	$0.22 \times 0.20 \times 0.18 \text{ mm}$
$Z = 8$	

### Data collection

Bruker SMART CCD area-detector diffractometer	5930 independent reflections
$\varphi$ and $\omega$ scans	4119 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.039$
$T_{\text{min}} = 0.754$ , $T_{\text{max}} = 0.833$	$\theta_{\text{max}} = 25.0^\circ$
17189 measured reflections	$h = -31 \rightarrow 31$
	$k = -10 \rightarrow 14$
	$l = -24 \rightarrow 27$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2 + 0.4655P]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.126$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.80 \text{ e \AA}^{-3}$
5930 reflections	$\Delta\rho_{\text{min}} = -0.78 \text{ e \AA}^{-3}$
429 parameters	
H-atom parameters constrained	

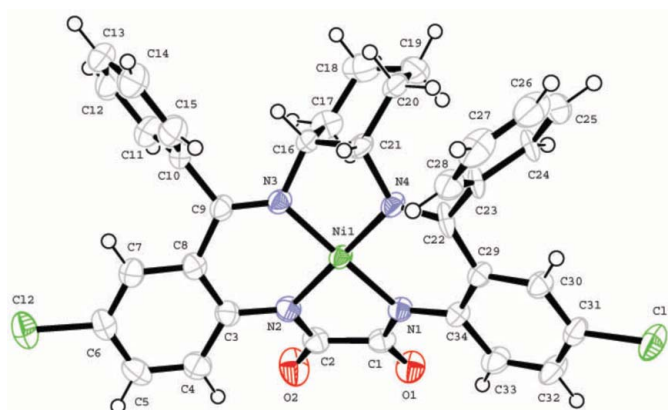
**Table 1**

Hydrogen-bond geometry (Å, °).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
C18—H18A...C11 <sup>i</sup>	0.97	2.93	3.764 (5)	145
C20—H20A...C11 <sup>i</sup>	0.97	2.85	3.720 (5)	150
C26—H26...O1 <sup>ii</sup>	0.93	2.61	3.452 (6)	151
C27—H27...O2 <sup>ii</sup>	0.93	2.49	3.223 (5)	136
C35—H35A...O2 <sup>ii</sup>	0.97	2.56	3.395 (7)	145

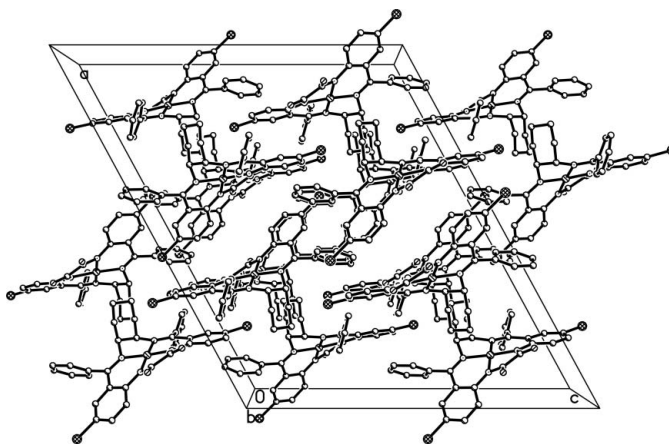
Symmetry codes: (i)  $x, -y + 1, z - \frac{1}{2}$  (ii)  $x, y + 1, z$ .

Two molecules of dichloromethane solvent were found in difference Fourier maps. The C atom of one of these molecules lies on a twofold axis. H atoms were placed in calculated positions and treated



**Figure 1**

A perspective view of the complex [NiL], showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.



**Figure 2**

Packing of the complex [NiL]·1.5(CH<sub>2</sub>Cl<sub>2</sub>). H atoms have been omitted.

as riding (C—H = 0.93–0.98 Å), with  $U_{\text{iso}}(\text{H})$  values set equal to  $1.2U_{\text{eq}}(\text{C})$ .

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXTL.

## References

- Bruker (1997). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2000). SHELXTL. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chen, C. T. & Suslick, K. S. (1993). *Coord. Chem. Rev.* **128**, 293–322.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Kahn, O. (1993). *Molecular Magnetism*. New York: VCH.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Vigato, P. A., Tamburini, S. & Fenton, D. E. (1990). *Coord. Chem. Rev.* **106**, 25–170.
- Wang, S.-B., Yang, G.-M., Liao, D.-Z. & Li, L.-C. (2004). *Inorg. Chem.* **43**, 852–854.
- Zhang, L., Wang, S.-B., Yang, G.-M., Tang, J.-K., Liao, D.-Z., Jiang, Z.-H., Yan, S.-P. & Cheng, P. (2003). *Inorg. Chem.* **42**, 1462–1466.
- Zhang, R.-H., Wang, Q.-L., Wang, Y.-F., Yang, G.-M., Liu, Z.-Q., Ding, B. & Xu, G.-F. (2005). *J. Coord. Chem.* **58**, 857–862.