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(7,12-Diphenyl-5,6:13,14-dibenzo-8,11-diazonia-1,4-diazacyclopentadeca-7,12-diene-2,3-dione)-nickel(II)

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.091
Data-to-parameter ratio = 16.0

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

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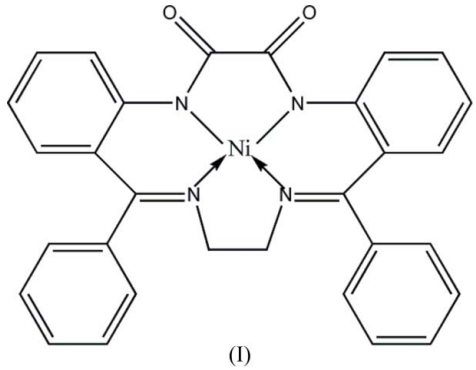
The title complex, $[Ni(C_{30}H_{22}N_4O_2)]$, has a square-planar geometry at the Ni atom.

Comment

Among the transition metal complexes of macrocyclic ligands (Wang *et al.*, 2004; Zhang *et al.*, 2003), the oxamide bridge serves as a pathway through which electron spin interaction takes place. The title complex, (I), has such a bridge (Fig. 1). As part of our ongoing investigation of macrocyclic complexes, we recently prepared (I). The Ni^{II} centre has a slightly distorted square-planar coordination formed by the four N atoms. The deviations of the four donors from their mean plane are 0.1225 (N1), −0.1245 (N2), 0.1228 (N3) and −0.1207 Å (N4), respectively, and the central Ni is −0.0352 Å out of the plane. Selected bond lengths and bond angles are listed in Table 1. Bond dimensions involving the Ni atom are comparable to those found in related compounds (Zhang *et al.*, 2006; Wang *et al.*, 2004).



(I)

Experimental

The compound was synthesized from the reaction of the macrocycle 2,2′-(oxalyldiimino)bis(benzophenone) (4.48 g, 0.01 mol), 1,2-ethanediamine (0.6 g, 0.01 mol) and $Ni(OAc)_2{\cdot}H_2O$ (2.48 g, 0.01 mol) for 7 h in methanol (50 ml) in the presence of NaOH (0.8 g). The mixture was cooled and filtered. The filtrate was kept at room temperature for several days to give red crystals.

Crystal data

$[Ni(C_{30}H_{22}N_4O_2)]$
M_r = 529.23
Monoclinic, $P2_1/c$
a = 15.817 (5) Å
b = 10.363 (3) Å
c = 16.321 (5) Å
β = 118.561 (3)°
V = 2349.7 (11) Å^3
Z = 4
D_x = 1.496 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.86 mm^{-1}
T = 294 (2) K
Block, red
0.06 × 0.04 × 0.04 mm

Data collection

Rigaku Saturn diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.950, T_{max} = 0.966
14754 measured reflections
5373 independent reflections
4318 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.091
S = 1.06
5373 reflections
335 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 0.3465P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N2	1.8527 (16)	Ni1—N4	1.8660 (17)
Ni1—N1	1.8572 (17)	Ni1—N3	1.8682 (17)
N2—Ni1—N1	86.44 (7)	C18—N1—Ni1	127.04 (14)
N2—Ni1—N4	174.58 (8)	C2—N2—Ni1	112.80 (13)
N1—Ni1—N4	94.58 (7)	C3—N2—Ni1	124.55 (14)
N2—Ni1—N3	92.66 (7)	C9—N3—Ni1	129.54 (14)
N1—Ni1—N3	170.18 (8)	C10—N3—Ni1	107.93 (13)
N4—Ni1—N3	87.23 (7)	C12—N4—Ni1	127.99 (15)
C1—N1—Ni1	110.49 (13)	C11—N4—Ni1	111.92 (12)

H atoms were included in calculated positions and refined as riding, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2005); software used to prepare material for publication: *CrystalStructure*.

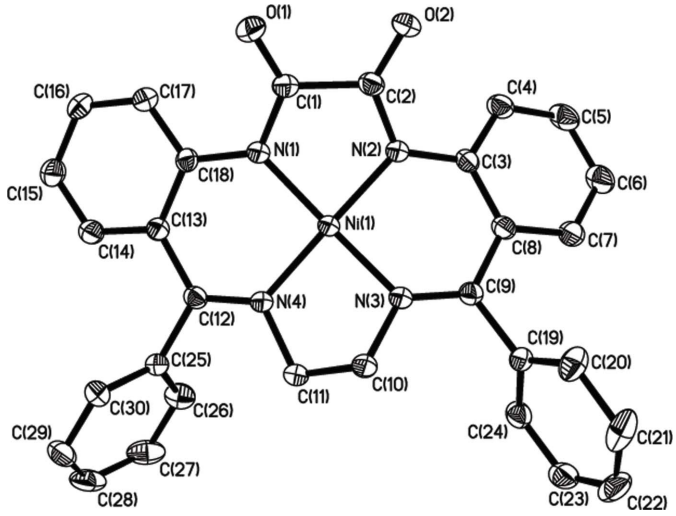


Figure 1
The molecular structure of (I) with 30% probability displacement ellipsoids. H atoms have been omitted.

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