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[2,3-Dioxo-5,6:14,15-dibenzo-1,4,8,12-tetraazacyclopentadeca-7,13-dienato(2–)]-nickel(II)

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Key indicators

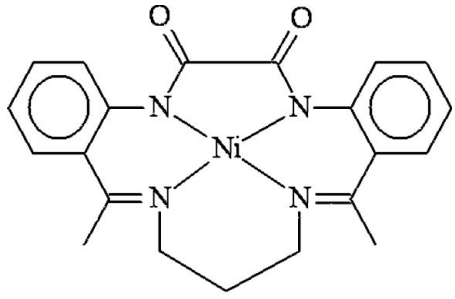
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.033
wR factor = 0.071
Data-to-parameter ratio = 12.3

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

In the title compound, [Ni($C_{21}H_{20}N_4O_2$)], the central Ni^{II} atom is coordinated by four N atoms, providing a slightly distorted square-planar environment with Ni—N distances ranging from 1.863 (3) to 1.912 (3) Å.

Comment

Macrocyclic oxamide complexes have been of great interest to supramolecular and coordination chemists due to the useful properties of the oxamide group. It can be coordinated to various metal ions and can influence an *exo–cis* conformation of the macrocyclic ring, which is suitable for the design of heterometallic systems (Wang *et al.*, 2004). Continuing our work on polynuclear macrocyclic complexes, we recently prepared the title Ni complex, (I), and present its crystal structure here (Fig. 1).



(I)

In compound (I), the Ni^{II} centre has a slightly distorted square-planar coordination formed by four N atoms, *viz.* N1 and N2 of the deprotonated oxamide and N3 and N4 from the 1,3-propanediamine. The deviations of the four N atoms from their least-squares plane are −0.2154 (14), 0.2081 (14), −0.1937 (13) and 0.2011 (14) Å, respectively. The central Ni atom is −0.0797 (9) Å out of this plane. The Ni—N bond distances range from 1.863 (3) to 1.912 (3) Å, in agreement with those of a related compound (Wang *et al.*, 2005).

The crystal packing is governed by van der Waals interactions only.

Experimental

The title compound was prepared by refluxing and stirring 2,2′-(oxalyldiimino)bis(benzophenone) (1 mmol), $Ni(OAc)_2·H_2O$ (1 mmol) and 1,3-propanediamine (2 mmol) for about 5 h in CH_3OH (50 ml) in the presence of 2 ml of 2 *M* NaOH. The mixture was cooled and filtered. Dark-red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of the filtrate in the open air at room temperature after two weeks.

Crystal data

$[Ni(C_{21}H_{20}N_4O_2)]$	$Z = 4$
$M_r = 419.12$	$D_x = 1.565$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 8.3453$ (14) Å	$\mu = 1.12$ mm^{-1}
$b = 10.9683$ (19) Å	$T = 294$ (2) K
$c = 19.436$ (3) Å	Block, red
$V = 1779.1$ (5) Å^3	0.24 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer	9174 measured reflections
φ and ω scans	3133 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2697 reflections with $I > 2\sigma(I)$
$T_{min} = 0.775$, $T_{max} = 0.842$	$R_{int} = 0.047$
	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.033$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.071$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.43$ e Å^{-3}
3133 reflections	Absolute structure: Flack (1983), 1320 Freidel pairs
255 parameters	Flack parameter: −0.007 (15)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Ni1—N1	1.863 (3)	Ni1—N3	1.888 (3)
Ni1—N2	1.873 (3)	Ni1—N4	1.912 (3)
N1—Ni1—N2	86.31 (11)	C21—N1—Ni1	123.0 (2)
N1—Ni1—N3	172.33 (12)	C2—N2—Ni1	110.0 (2)
N2—Ni1—N3	94.29 (11)	C3—N2—Ni1	127.3 (2)
N1—Ni1—N4	89.74 (11)	C9—N3—Ni1	127.7 (2)
N2—Ni1—N4	161.94 (11)	C11—N3—Ni1	113.3 (2)
N3—Ni1—N4	91.96 (12)	C14—N4—Ni1	127.8 (3)
C1—N1—Ni1	113.5 (2)	C13—N4—Ni1	109.8 (2)

H atoms bonded to C atoms were included in calculated positions and refined as riding, 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, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

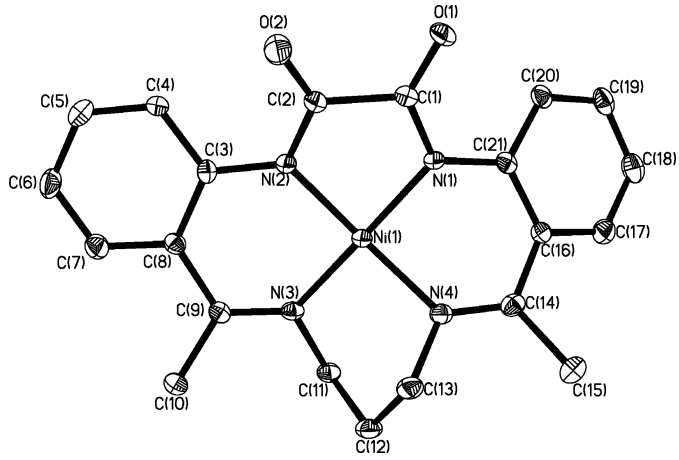


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted.

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