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[5,6:14,15-Dibenzo-1,4,8,12-tetraazacyclopentadeca-7,13-diene-2,3-dione(2–)]nickel(II) monohydrate

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The nickel(II) ion in the title complex, $[Ni(C_{19}H_{16}N_4O_2)]\cdot H_2O$, is coordinated by four N atoms from the 5,6:14,15-dibenzo-1,4,8,12-tetraazacyclopentadeca-7,13-diene-2,3-dione(2−) ligand in chelating mode, forming a distorted square-planar geometry. The complex molecules are associated into a two-dimensional superamolecular architecture through weak intermolecular O—H···O and C—H···O hydrogen bonding.

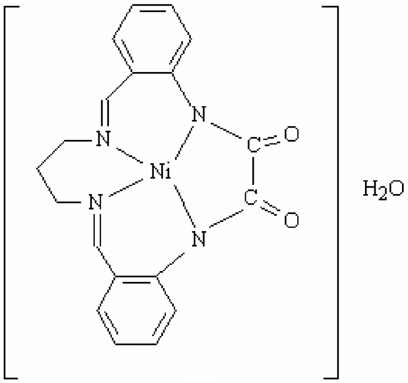
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.070
Data-to-parameter ratio = 14.1

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

Comment

The coordination properties of *N*,*N*′-trisoxamides have been thoroughly investigated both in aqueous solution and in the solid state (Ruiz *et al.*, 1999). The easy *cis–trans* isomerization equilibria that they exhibit, together with the great variety of *N*,*N*′-trisoxamides that can be used to vary the overall charge, complexing ability and polarity, make them very suitable ligands in designing homo- and heterometallic species. Furthermore, in the presence of metal ions and when the oxamide has another coordinating group at a position that can form five- or six-membered chelate rings, the amide group deproronates and coordinates simultaneously in a low pH range (Ojima & Nakamoto, 1980). Recently, increasing attention has been paid to the design of mononuclear oxamide complexes of ligands containing macrocyclic rings, because these precursors are particularly suitable for designing heterometallic complexes (Nakatani *et al.*, 1989; Lloret *et al.*, 1993). However, most of the studies have been focused on the mononuclear oxamide copper complexes, while the mononuclear oxamide nickel complex has been poorly investigated (Andruh *et al.*, 1993; Bencini *et al.*, 1986). We therefore designed and synthesized the macrocyclic oxamide title complex, (I).



(I)

The structure of (I) consists of the neutral Ni^{II} complex of the [15] N4 macrocyclic oxamide and water (Fig. 1). The

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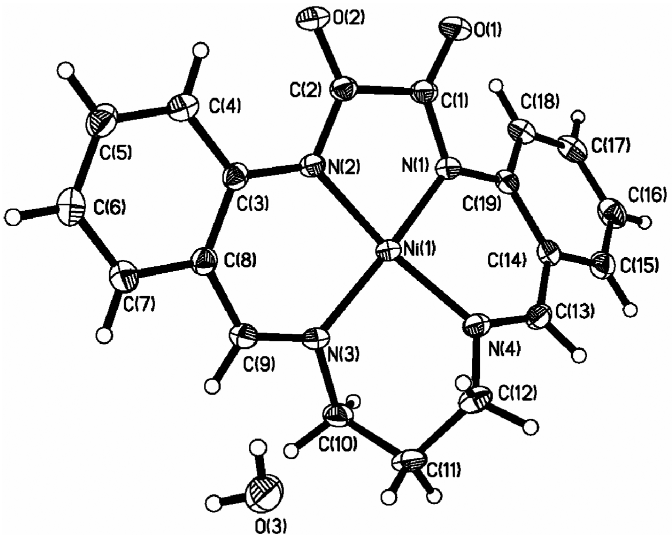

Figure 1
An *ORTEPIII* (Burnett & Johnson, 1996) view of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 70% probability level.

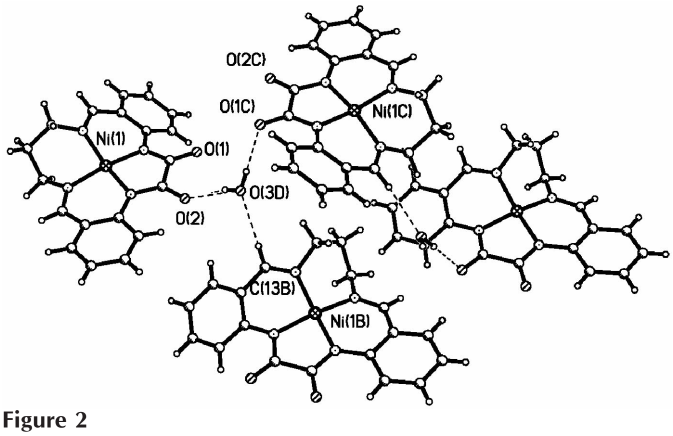

Figure 2
View of hydrogen-bond interactions (dashed lines) in the complex. [Symmetry codes: (*B*) $-\frac{1}{2}+x, -\frac{1}{2}-y, \frac{1}{2}+z$; (*C*) $-x, -y, -z+1$; (*D*) $-x, -y+1, -z+1$.]

macrocyclic ligand coordinates to the Ni^{II} ion *via* two deprotonated oxamide N atoms and two imine N atoms. The Ni^{II} ion is displaced from the N_4 least-squares plane by 0.0084 (2) Å, while the deviations of the four N-atom donors (N1, N2, N3 and N4) from this plane are 0.2609 (2), −0.2602 (3), 0.2503 (2) and −0.2594 (4) Å, respectively, showing that the NiN_4 chromophore assumes a planar coordination geometry. Fig. 2 indicates that O–H···O and C–H···O hydrogen-bond interactions link the mononuclear fragments and water to form a two-dimensional superamolecular architecture; the hydrogen-bond distances O3*D*–H3*AD*···O1*C* and O3*D*–H3*BD*···O2 are 2.932 (2) and 2.841 (4) Å, respectively, while the hydrogen-bond distance C13*B*–H13*B*···O3*D* is 3.317 (2) Å (see Fig. 2).

Experimental

The title compound was prepared by refluxing and stirring diformyloxanilide (2.49 g, 0.01 mol), 1,3-propanediamine (1.6 ml, 0.02 mol) and nickel(II) perchlorate (0.01 mol) for 3.5 h in 50 ml of MeOH, in the presence of four drops of 2 *M* NaOH. After the mixture was cooled and filtered, the resulting deep-red filtrate, kept at room temperature for several days, gave rise to deep-red crystals suitable for X-ray analysis. Analysis: calculated for $C_{19}H_{18}N_4NiO_3$: C 55.79, H 4.43, N 13.70%; found: C 55.75, H 4.36, N 13.34%.

Crystal data

$[Ni(C_{19}H_{16}N_4O_2)]\cdot H_2O$
$M_r = 409.08$
Monoclinic, $P2_1/n$
$a = 8.335$ (3) Å
$b = 10.034$ (3) Å
$c = 20.017$ (6) Å
$\beta = 91.810$ (6)°
$V = 1673.2$ (9) Å^3
$Z = 4$
$D_x = 1.624$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1013 reflections
$\theta = 2.7$–26.4°
$\mu = 1.19$ mm^{-1}
$T = 293$ (2) K
Block, red
0.26 × 0.24 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.694$, $T_{max} = 0.770$
9419 measured reflections
3446 independent reflections
2772 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 26.4°$
$h = -7 \rightarrow 10$
$k = -10 \rightarrow 12$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.070$
$S = 1.03$
3446 reflections
245 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2 + 0.5705P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0106 (6)

Table 1
Selected geometric parameters (Å, °).

Ni1—N3	1.8744 (17)	Ni1—N4	1.8934 (17)
Ni1—N1	1.8792 (18)	Ni1—N2	1.9042 (16)
N3—Ni1—N1	164.86 (7)	N3—Ni1—N2	94.84 (7)
N3—Ni1—N4	89.61 (7)	N1—Ni1—N2	86.93 (7)
N1—Ni1—N4	92.90 (7)	N4—Ni1—N2	163.58 (7)

Water H atoms were located in difference Fourier maps and constrained as riding atoms with O—H distances set at 0.85 Å. Other H atoms were constrained as riding atoms, with C—H distances set at 0.93 and 0.97 Å. U_{iso}(H) values were set at $1.2U_{eq}$(C) and $1.5U_{eq}$(O).

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-NT* (Bruker, 1998); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnston, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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