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Diaqua[*N*,*N*′-bis(4-methoxybenzyl)-2,2′-(ethane-1,2-diyldiimino)diacetato]-nickel(II) sesquihydrate

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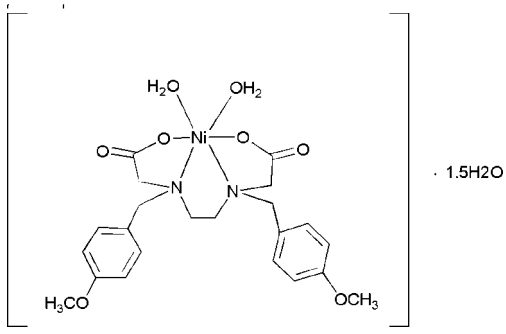
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; disorder in solvent or counterion; R factor = 0.043; wR factor = 0.133; data-to-parameter ratio = 16.1.

In the title compound, $[Ni(C_{22}H_{26}N_2O_6)(H_2O)_2]\cdot 1.5H_2O$, the Ni^{II} ion is in a slightly distorted octahedral coordination enviroment formed by an N_2O_4 donor set. In the crystal structure, intermolecular O—H···O and C—H···O hydrogen bonds and C—H···π interactions connect molecules into a three-dimensional network.

Related literature

For related literature, see: Zhang, Lan *et al.* (2007); Zhang, Weng, Hu *et al.* (2007); Zhang, Weng & Xu (2007); Xu *et al.* (2004).



Experimental

Crystal data

$[Ni(C_{22}H_{26}N_2O_6)(H_2O)_2]\cdot 1.5H_2O$
M_r = 536.20
Monoclinic, $C2/c$
a = 25.1393 (14) Å
b = 8.1256 (4) Å
c = 26.6925 (15) Å
β = 114.248 (2)°
V = 4971.5 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.84 mm^{-1}
T = 294 (2) K
0.30 × 0.23 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
26930 measured reflections
5429 independent reflections
4279 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.133
S = 1.03
5429 reflections
337 parameters
9 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni—O7	2.0352 (15)	Ni—O8	2.0818 (15)
Ni—O1	2.0529 (14)	Ni—N2	2.1159 (18)
Ni—O4	2.0666 (14)	Ni—N1	2.1212 (17)

Table 2
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C16–C21 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7*A*···O4^{i}	0.791 (15)	1.984 (16)	2.769 (2)	172 (3)
O7—H7*B*···O1^{ii}	0.757 (15)	1.900 (16)	2.656 (2)	176 (2)
O8—H8*B*···O2^{ii}	0.776 (17)	2.027 (16)	2.787 (2)	166 (3)
O10—H10*A*···O3^{iii}	0.784 (17)	2.076 (17)	2.857 (2)	174 (3)
O10—H10*B*···O5^{iv}	0.796 (17)	2.12 (2)	2.851 (3)	152 (3)
C5—H5*B*···O9^{v}	0.97	2.49	3.430 (5)	162
C15—H15*B*···O9	0.97	2.52	3.451 (5)	161
O8—H8*A*···O10	0.832 (16)	1.913 (17)	2.731 (3)	167 (3)
C12—H12*C*···*Cg*1^{vi}	0.96	2.80	3.746 (3)	169

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2570).

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  doi:10.1107/S1600536807060849

supplementary materials

Acta Cryst. (2007). E**63**, m3164 [**doi:10.1107/S1600536807060849**]

Diaqua[*N*,*N*'-bis(4-methoxybenzyl)-2,2'-(ethane-1,2-diyldiimino)diacetato]nickel(II) sesquihydrate

M. Zhang, X. Song, P. Su and X. Meng

Comment

We have previously synthesized and characterized Ni, Zn and Cd complexes using the ligand [N, *N*'-bis(4-methoxylbenzyl) ethane −1,2-diyldiimino] diacetate (H_2L) (Zhang, Lan *et al.*, 2007; Zhang, Weng, Hu *et al.*, 2007; Zhang, Weng & Xu 2007; Xu *et al.*, 2004). As part of our continuing studies of these types of complexes, we report the crystal structure of the title complex (I).

In (I), the Ni^{II} ion is in a slightly distorted octahedral coordination environment formed by an N_2O_4 donor set (Fig.1). There are no significant differences between the bond lengths and angles around the central nickel atom in and that of earlier reported analogs. In the crystal structure (Fig 2) O—H···O, C—H···O hydrogen bonds and C—H···π interaction, link molecules into a three-dimensional network.

Experimental

The ligand [*N*,*N*'-bis(4-methoxylbenzyl)ethane-1,2-diyldiimino] diacetate(H_2L) and its nickel complex were prepared according to the literature method (Xu *et al.*, 2004). Crystals were obtained by slow evaporation (three weeks) of a methanol solution (15 ml) of the title complex (0.046 g, 0.1 mmol).

Refinement

H atoms bonded to C atoms were positioned geometrically with C—H = 0.93 Å(aromatic), 0.97Å (methylene) and 0.96Å (methyl) and with U_{iso}(H) = kU_{eq}(C) (k = 1.5 for methyl and 1.2 for the other C atoms. H atoms bonded to water O7, O8 and O10 atoms were found in the difference maps and refined with U_{iso}(H)= 1.5U_{eq}(O). Water atom O9, refined with a partial occupancy of 0.516 (1) but this was eventually fixed at 0.5. H atoms attached to O9 were included in their as found positions with U_{iso}(H) = 1.5U_{eq}(O).

Figures

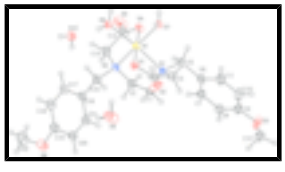

Fig. 1. The molecular structure of showing 30% probability displacement ellipsoids.

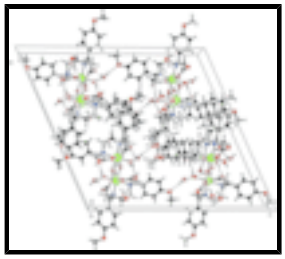

Fig. 2. Part of the crystal structure showing the formation of a three-dimensional network. Dashed lines denote hydrogen bonds.

Diaqua[*N*,*N*'-bis(4-methoxybenzyl)-2,2'-(ethane-1,2- diyldiimino)diacetato]nickel(II) sesquihydrate

Crystal data

$[Ni(C_{22}H_{26}N_2O_6)(H_2O)_2]\cdot 1.5H_2O$	F_{000} = 2264
M_r = 536.20	D_x = 1.446 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 5321 reflections
a = 25.1393 (14) Å	θ = 1.6–26.9º
b = 8.1256 (4) Å	μ = 0.84 mm^{-1}
c = 26.6925 (15) Å	*T* = 294 (2) K
β = 114.248 (2)º	Block, blue
V = 4971.5 (5) Å^3	0.30 × 0.23 × 0.20 mm
Z = 8	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	4279 reflections with $I > 2\sigma(I)$
Radiation source: fine focus sealed Siemens Mo tube	R_{int} = 0.056
Monochromator: graphite	θ_{max} = 27.0º
T = 294(2) K	θ_{min} = 1.7º
0.3° wide ω exposures scans	*h* = −32→31
Absorption correction: none	*k* = −10→10
26930 measured reflections	*l* = −33→34
5429 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.133	$w = 1/[\sigma^2(F_o^2) + (0.0925P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
5429 reflections	$\Delta\rho_{max}$ = 0.45 e Å^{-3}

337 parameters
9 restraints
Primary atom site location: structure-invariant direct methods

$\Delta\rho_{min} = -0.36$ e Å^{-3}
Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ni	0.177788 (10)	0.49923 (3)	0.223662 (10)	0.03190 (12)	
C1	0.08623 (9)	0.5097 (2)	0.26158 (10)	0.0371 (5)	
H1A	0.0816	0.6283	0.2595	0.044*	
H1B	0.0669	0.4671	0.2838	0.044*	
C2	0.05859 (9)	0.4373 (3)	0.20478 (9)	0.0388 (5)	
H2A	0.0611	0.3182	0.2072	0.047*	
H2B	0.0176	0.4673	0.1879	0.047*	
C3	0.18198 (9)	0.5916 (3)	0.32888 (8)	0.0384 (5)	
H3A	0.2204	0.5493	0.3520	0.046*	
H3B	0.1616	0.6145	0.3521	0.046*	
C4	0.18819 (9)	0.7496 (3)	0.30125 (9)	0.0395 (5)	
C5	0.16049 (9)	0.2968 (3)	0.31155 (9)	0.0384 (5)	
H5A	0.2017	0.2727	0.3239	0.046*	
H5B	0.1393	0.2194	0.2825	0.046*	
C6	0.14326 (9)	0.2674 (3)	0.35899 (9)	0.0375 (5)	
C7	0.08884 (9)	0.2062 (3)	0.35051 (9)	0.0406 (5)	
H7	0.0621	0.1847	0.3148	0.049*	
C8	0.07307 (10)	0.1761 (3)	0.39378 (9)	0.0406 (5)	
H8	0.0363	0.1348	0.3871	0.049*	
C9	0.11248 (10)	0.2080 (3)	0.44652 (9)	0.0472 (6)	
C10	0.16688 (11)	0.2712 (4)	0.45653 (11)	0.0611 (8)	
H10	0.1931	0.2955	0.4922	0.073*	
C11	0.18194 (10)	0.2981 (4)	0.41266 (10)	0.0549 (7)	
H11	0.2190	0.3378	0.4195	0.066*	
C12	0.05006 (11)	0.0964 (4)	0.48656 (11)	0.0599 (7)	
H12A	0.0169	0.1655	0.4674	0.090*	
H12B	0.0516	0.0699	0.5222	0.090*	
H12C	0.0466	−0.0030	0.4661	0.090*	
C13	0.08348 (9)	0.3750 (3)	0.12824 (9)	0.0415 (5)	

H13A	0.0920	0.4271	0.0997	0.050*	
H13B	0.0438	0.3336	0.1116	0.050*	
C14	0.12553 (9)	0.2309 (3)	0.15258 (10)	0.0423 (5)	
C15	0.06626 (9)	0.6627 (3)	0.14556 (9)	0.0392 (5)	
H15A	0.0872	0.6943	0.1236	0.047*	
H15B	0.0761	0.7420	0.1752	0.047*	
C16	0.00181 (9)	0.6764 (3)	0.11018 (9)	0.0394 (5)	
C17	−0.02254 (10)	0.6239 (4)	0.05617 (9)	0.0529 (6)	
H17	0.0016	0.5790	0.0411	0.063*	
C18	−0.08187 (11)	0.6362 (4)	0.02370 (10)	0.0601 (7)	
H18	−0.0972	0.5967	−0.0122	0.072*	
C19	−0.11784 (10)	0.7070 (3)	0.04483 (11)	0.0532 (6)	
C20	−0.09445 (10)	0.7631 (3)	0.09848 (10)	0.0481 (6)	
H20	−0.1185	0.8121	0.1129	0.058*	
C21	−0.03585 (10)	0.7467 (3)	0.13052 (9)	0.0426 (5)	
H21	−0.0209	0.7835	0.1667	0.051*	
C22	−0.20177 (13)	0.6894 (6)	−0.04023 (13)	0.1024 (14)	
H22A	−0.1866	0.7626	−0.0594	0.154*	
H22B	−0.2434	0.7019	−0.0543	0.154*	
H22C	−0.1925	0.5779	−0.0455	0.154*	
N1	0.14956 (7)	0.4672 (2)	0.28780 (7)	0.0312 (4)	
N2	0.08809 (8)	0.49765 (18)	0.17022 (8)	0.0331 (4)	
O1	0.18227 (6)	0.73640 (17)	0.25161 (6)	0.0361 (3)	
O2	0.19946 (9)	0.8785 (2)	0.32799 (8)	0.0633 (5)	
O3	0.10204 (8)	0.1810 (3)	0.49259 (7)	0.0660 (6)	
O4	0.16957 (6)	0.25788 (17)	0.19711 (6)	0.0363 (3)	
O5	0.11420 (8)	0.0997 (2)	0.12718 (8)	0.0753 (6)	
O6	−0.17656 (7)	0.7273 (3)	0.01645 (9)	0.0784 (7)	
O7	0.26310 (6)	0.47501 (19)	0.27647 (6)	0.0326 (3)	
H7A	0.2796 (10)	0.560 (2)	0.2818 (11)	0.049*	
H7B	0.2772 (10)	0.405 (2)	0.2679 (10)	0.049*	
O8	0.20131 (7)	0.5574 (2)	0.15954 (7)	0.0425 (4)	
H8B	0.2297 (10)	0.519 (3)	0.1597 (13)	0.064*	
H8A	0.1971 (12)	0.653 (2)	0.1477 (11)	0.064*	
O10	0.18169 (8)	0.8511 (2)	0.10513 (7)	0.0541 (4)	
H10A	0.1592 (12)	0.836 (4)	0.0748 (8)	0.081*	
H10B	0.1741 (14)	0.932 (3)	0.1176 (12)	0.081*	
O9	0.0732 (2)	0.9932 (4)	0.2293 (2)	0.0868 (16)	0.50
H9A	0.1124	0.9932	0.2293	0.130*	0.50
H9B	0.0502	0.9984	0.1962	0.130*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni	0.02839 (18)	0.03414 (19)	0.03438 (18)	0.00004 (9)	0.01411 (13)	0.00305 (9)
C1	0.0288 (11)	0.0420 (13)	0.0468 (12)	0.0068 (8)	0.0222 (10)	0.0130 (9)
C2	0.0260 (10)	0.0412 (12)	0.0497 (12)	−0.0009 (9)	0.0161 (9)	0.0099 (10)
C3	0.0419 (12)	0.0422 (13)	0.0375 (10)	0.0029 (9)	0.0226 (9)	0.0001 (9)

C4	0.0372 (11)	0.0392 (12)	0.0495 (12)	0.0011 (9)	0.0253 (10)	−0.0020 (10)
C5	0.0367 (11)	0.0394 (12)	0.0442 (11)	0.0068 (9)	0.0216 (9)	0.0133 (9)
C6	0.0357 (10)	0.0391 (12)	0.0402 (11)	0.0039 (9)	0.0180 (9)	0.0125 (9)
C7	0.0388 (11)	0.0430 (13)	0.0394 (11)	−0.0021 (9)	0.0155 (9)	0.0066 (9)
C8	0.0370 (11)	0.0430 (13)	0.0447 (12)	−0.0031 (9)	0.0198 (10)	0.0070 (10)
C9	0.0458 (13)	0.0592 (16)	0.0408 (12)	−0.0032 (11)	0.0221 (10)	0.0111 (11)
C10	0.0466 (14)	0.091 (2)	0.0383 (12)	−0.0185 (14)	0.0104 (10)	0.0138 (13)
C11	0.0364 (12)	0.0805 (19)	0.0439 (12)	−0.0084 (12)	0.0126 (10)	0.0180 (12)
C12	0.0507 (15)	0.086 (2)	0.0526 (14)	−0.0012 (14)	0.0313 (12)	0.0142 (14)
C13	0.0344 (11)	0.0452 (13)	0.0367 (10)	0.0014 (9)	0.0063 (9)	0.0016 (9)
C14	0.0348 (11)	0.0406 (13)	0.0464 (12)	−0.0011 (9)	0.0116 (9)	−0.0044 (10)
C15	0.0335 (11)	0.0382 (12)	0.0449 (11)	0.0045 (9)	0.0151 (9)	0.0132 (9)
C16	0.0348 (11)	0.0415 (12)	0.0416 (11)	0.0070 (9)	0.0154 (9)	0.0128 (9)
C17	0.0433 (13)	0.0733 (18)	0.0426 (12)	0.0167 (12)	0.0181 (10)	0.0102 (12)
C18	0.0486 (14)	0.085 (2)	0.0395 (12)	0.0121 (14)	0.0111 (11)	0.0049 (13)
C19	0.0351 (12)	0.0685 (18)	0.0529 (14)	0.0058 (11)	0.0147 (11)	0.0116 (12)
C20	0.0387 (12)	0.0538 (15)	0.0579 (14)	0.0095 (10)	0.0259 (11)	0.0125 (12)
C21	0.0418 (12)	0.0451 (13)	0.0419 (11)	0.0041 (10)	0.0183 (10)	0.0077 (10)
C22	0.0467 (17)	0.175 (4)	0.0621 (19)	0.011 (2)	−0.0008 (15)	0.016 (2)
N1	0.0263 (8)	0.0344 (9)	0.0362 (9)	0.0035 (7)	0.0162 (7)	0.0067 (7)
N2	0.0276 (9)	0.0344 (10)	0.0373 (9)	0.0029 (6)	0.0132 (8)	0.0078 (7)
O1	0.0374 (8)	0.0330 (8)	0.0426 (8)	−0.0016 (6)	0.0210 (6)	0.0019 (6)
O2	0.0919 (14)	0.0448 (10)	0.0778 (12)	−0.0143 (9)	0.0598 (11)	−0.0192 (9)
O3	0.0639 (11)	0.0989 (15)	0.0404 (9)	−0.0248 (11)	0.0267 (8)	0.0083 (9)
O4	0.0310 (7)	0.0343 (8)	0.0388 (8)	0.0017 (6)	0.0097 (6)	0.0007 (6)
O5	0.0577 (11)	0.0523 (12)	0.0821 (13)	0.0061 (9)	−0.0056 (10)	−0.0270 (10)
O6	0.0323 (9)	0.126 (2)	0.0665 (12)	0.0121 (10)	0.0096 (9)	0.0082 (12)
O7	0.0276 (8)	0.0343 (8)	0.0361 (8)	0.0007 (6)	0.0134 (6)	−0.0032 (6)
O8	0.0422 (9)	0.0480 (9)	0.0447 (9)	0.0053 (8)	0.0254 (7)	0.0098 (8)
O10	0.0628 (11)	0.0548 (11)	0.0409 (9)	0.0101 (9)	0.0176 (8)	0.0019 (8)
O9	0.066 (3)	0.066 (3)	0.115 (4)	0.0022 (19)	0.023 (3)	−0.020 (2)

Geometric parameters (Å, °)

Ni—O7	2.0352 (15)	C12—H12B	0.9600
Ni—O1	2.0529 (14)	C12—H12C	0.9600
Ni—O4	2.0666 (14)	C13—N2	1.468 (3)
Ni—O8	2.0818 (15)	C13—C14	1.532 (3)
Ni—N2	2.1159 (18)	C13—H13A	0.9700
Ni—N1	2.1212 (17)	C13—H13B	0.9700
C1—N1	1.493 (3)	C14—O5	1.232 (3)
C1—C2	1.504 (3)	C14—O4	1.267 (3)
C1—H1A	0.9700	C15—N2	1.496 (3)
C1—H1B	0.9700	C15—C16	1.508 (3)
C2—N2	1.484 (3)	C15—H15A	0.9700
C2—H2A	0.9700	C15—H15B	0.9700
C2—H2B	0.9700	C16—C17	1.382 (3)
C3—N1	1.468 (3)	C16—C21	1.393 (3)
C3—C4	1.521 (3)	C17—C18	1.387 (3)

C3—H3A	0.9700	C17—H17	0.9300
C3—H3B	0.9700	C18—C19	1.373 (4)
C4—O2	1.233 (3)	C18—H18	0.9300
C4—O1	1.276 (3)	C19—O6	1.364 (3)
C5—N1	1.500 (3)	C19—C20	1.383 (4)
C5—C6	1.516 (3)	C20—C21	1.373 (3)
C5—H5A	0.9700	C20—H20	0.9300
C5—H5B	0.9700	C21—H21	0.9300
C6—C11	1.384 (3)	C22—O6	1.413 (4)
C6—C7	1.384 (3)	C22—H22A	0.9600
C7—C8	1.388 (3)	C22—H22B	0.9600
C7—H7	0.9300	C22—H22C	0.9600
C8—C9	1.373 (3)	O7—H7A	0.791 (15)
C8—H8	0.9300	O7—H7B	0.757 (15)
C9—O3	1.376 (3)	O8—H8B	0.776 (17)
C9—C10	1.380 (3)	O8—H8A	0.832 (16)
C10—C11	1.388 (4)	O10—H10A	0.784 (17)
C10—H10	0.9300	O10—H10B	0.796 (17)
C11—H11	0.9300	O9—H9A	0.9830
C12—O3	1.426 (3)	O9—H9B	0.8345
C12—H12A	0.9600		
O7—Ni—O1	87.37 (6)	H12B—C12—H12C	109.5
O7—Ni—O4	94.26 (6)	N2—C13—C14	111.77 (17)
O1—Ni—O4	176.70 (5)	N2—C13—H13A	109.3
O7—Ni—O8	90.98 (6)	C14—C13—H13A	109.3
O1—Ni—O8	95.39 (6)	N2—C13—H13B	109.3
O4—Ni—O8	87.46 (7)	C14—C13—H13B	109.3
O7—Ni—N2	173.93 (6)	H13A—C13—H13B	107.9
O1—Ni—N2	97.76 (6)	O5—C14—O4	125.5 (2)
O4—Ni—N2	80.46 (6)	O5—C14—C13	117.7 (2)
O8—Ni—N2	91.78 (7)	O4—C14—C13	116.77 (19)
O7—Ni—N1	92.06 (6)	N2—C15—C16	116.57 (18)
O1—Ni—N1	78.96 (6)	N2—C15—H15A	108.1
O4—Ni—N1	98.10 (6)	C16—C15—H15A	108.1
O8—Ni—N1	173.46 (6)	N2—C15—H15B	108.1
N2—Ni—N1	85.77 (7)	C16—C15—H15B	108.1
N1—C1—C2	110.15 (17)	H15A—C15—H15B	107.3
N1—C1—H1A	109.6	C17—C16—C21	116.9 (2)
C2—C1—H1A	109.6	C17—C16—C15	122.2 (2)
N1—C1—H1B	109.6	C21—C16—C15	120.9 (2)
C2—C1—H1B	109.6	C16—C17—C18	122.1 (2)
H1A—C1—H1B	108.1	C16—C17—H17	118.9
N2—C2—C1	110.81 (17)	C18—C17—H17	118.9
N2—C2—H2A	109.5	C19—C18—C17	119.6 (2)
C1—C2—H2A	109.5	C19—C18—H18	120.2
N2—C2—H2B	109.5	C17—C18—H18	120.2
C1—C2—H2B	109.5	O6—C19—C18	124.6 (2)
H2A—C2—H2B	108.1	O6—C19—C20	116.0 (2)
N1—C3—C4	110.80 (17)	C18—C19—C20	119.5 (2)

N1—C3—H3A	109.5	C21—C20—C19	120.3 (2)
C4—C3—H3A	109.5	C21—C20—H20	119.9
N1—C3—H3B	109.5	C19—C20—H20	119.9
C4—C3—H3B	109.5	C20—C21—C16	121.6 (2)
H3A—C3—H3B	108.1	C20—C21—H21	119.2
O2—C4—O1	125.0 (2)	C16—C21—H21	119.2
O2—C4—C3	118.8 (2)	O6—C22—H22A	109.5
O1—C4—C3	116.18 (19)	O6—C22—H22B	109.5
N1—C5—C6	115.65 (17)	H22A—C22—H22B	109.5
N1—C5—H5A	108.4	O6—C22—H22C	109.5
C6—C5—H5A	108.4	H22A—C22—H22C	109.5
N1—C5—H5B	108.4	H22B—C22—H22C	109.5
C6—C5—H5B	108.4	C3—N1—C1	110.43 (16)
H5A—C5—H5B	107.4	C3—N1—C5	111.74 (16)
C11—C6—C7	117.5 (2)	C1—N1—C5	112.73 (16)
C11—C6—C5	121.1 (2)	C3—N1—Ni	104.56 (12)
C7—C6—C5	121.5 (2)	C1—N1—Ni	103.84 (12)
C6—C7—C8	121.9 (2)	C5—N1—Ni	112.98 (12)
C6—C7—H7	119.1	C13—N2—C2	110.31 (16)
C8—C7—H7	119.1	C13—N2—C15	111.92 (17)
C9—C8—C7	119.2 (2)	C2—N2—C15	112.52 (17)
C9—C8—H8	120.4	C13—N2—Ni	104.29 (12)
C7—C8—H8	120.4	C2—N2—Ni	104.43 (12)
C8—C9—O3	124.3 (2)	C15—N2—Ni	112.83 (12)
C8—C9—C10	120.6 (2)	C4—O1—Ni	114.97 (13)
O3—C9—C10	115.1 (2)	C9—O3—C12	118.57 (19)
C9—C10—C11	119.1 (2)	C14—O4—Ni	114.31 (14)
C9—C10—H10	120.4	C19—O6—C22	118.4 (2)
C11—C10—H10	120.4	Ni—O7—H7A	111.5 (19)
C6—C11—C10	121.7 (2)	Ni—O7—H7B	110.9 (19)
C6—C11—H11	119.1	H7A—O7—H7B	116 (2)
C10—C11—H11	119.1	Ni—O8—H8B	118 (2)
O3—C12—H12A	109.5	Ni—O8—H8A	119.4 (19)
O3—C12—H12B	109.5	H8B—O8—H8A	110 (2)
H12A—C12—H12B	109.5	H10A—O10—H10B	112 (3)
O3—C12—H12C	109.5	H9A—O9—H9B	105.1
H12A—C12—H12C	109.5		
N1—C1—C2—N2	−58.1 (2)	O1—Ni—N1—C1	83.25 (12)
N1—C3—C4—O2	160.3 (2)	O4—Ni—N1—C1	−95.24 (12)
N1—C3—C4—O1	−21.0 (3)	N2—Ni—N1—C1	−15.52 (12)
N1—C5—C6—C11	−88.3 (3)	O7—Ni—N1—C5	−67.38 (14)
N1—C5—C6—C7	92.9 (3)	O1—Ni—N1—C5	−154.29 (14)
C11—C6—C7—C8	0.0 (3)	O4—Ni—N1—C5	27.22 (14)
C5—C6—C7—C8	178.8 (2)	N2—Ni—N1—C5	106.94 (14)
C6—C7—C8—C9	0.1 (4)	C14—C13—N2—C2	−76.0 (2)
C7—C8—C9—O3	−179.1 (2)	C14—C13—N2—C15	157.91 (18)
C7—C8—C9—C10	0.8 (4)	C14—C13—N2—Ni	35.6 (2)
C8—C9—C10—C11	−1.7 (5)	C1—C2—N2—C13	151.50 (17)
O3—C9—C10—C11	178.2 (3)	C1—C2—N2—C15	−82.7 (2)

C7—C6—C11—C10	−0.9 (4)	C1—C2—N2—Ni	39.97 (19)
C5—C6—C11—C10	−179.7 (3)	C16—C15—N2—C13	67.1 (2)
C9—C10—C11—C6	1.8 (5)	C16—C15—N2—C2	−57.8 (2)
N2—C13—C14—O5	158.0 (2)	C16—C15—N2—Ni	−175.64 (15)
N2—C13—C14—O4	−22.7 (3)	O1—Ni—N2—C13	153.15 (13)
N2—C15—C16—C17	−80.1 (3)	O4—Ni—N2—C13	−29.66 (13)
N2—C15—C16—C21	101.4 (3)	O8—Ni—N2—C13	57.46 (13)
C21—C16—C17—C18	−1.6 (4)	N1—Ni—N2—C13	−128.61 (13)
C15—C16—C17—C18	179.8 (2)	O1—Ni—N2—C2	−91.04 (13)
C16—C17—C18—C19	2.0 (4)	O4—Ni—N2—C2	86.15 (13)
C17—C18—C19—O6	179.1 (3)	O8—Ni—N2—C2	173.27 (13)
C17—C18—C19—C20	−0.9 (4)	N1—Ni—N2—C2	−12.80 (13)
O6—C19—C20—C21	179.4 (2)	O4—Ni—N2—C15	−151.34 (15)
C18—C19—C20—C21	−0.6 (4)	O8—Ni—N2—C15	−64.22 (15)
C19—C20—C21—C16	1.0 (4)	N1—Ni—N2—C15	109.71 (15)
C17—C16—C21—C20	0.1 (3)	O2—C4—O1—Ni	169.82 (19)
C15—C16—C21—C20	178.7 (2)	C3—C4—O1—Ni	−8.7 (2)
C4—C3—N1—C1	−73.8 (2)	O8—Ni—O1—C4	−159.23 (15)
C4—C3—N1—C5	159.87 (16)	N2—Ni—O1—C4	108.24 (15)
C4—C3—N1—Ni	37.33 (18)	N1—Ni—O1—C4	24.12 (14)
C2—C1—N1—C3	153.48 (17)	C8—C9—O3—C12	7.9 (4)
C2—C1—N1—C5	−80.7 (2)	C10—C9—O3—C12	−172.0 (3)
C2—C1—N1—Ni	41.88 (18)	O5—C14—O4—Ni	174.6 (2)
C6—C5—N1—C3	60.3 (2)	C13—C14—O4—Ni	−4.6 (3)
C6—C5—N1—C1	−64.7 (2)	O7—Ni—O4—C14	−162.88 (15)
C6—C5—N1—Ni	177.91 (14)	O8—Ni—O4—C14	−72.09 (15)
O7—Ni—N1—C3	54.35 (12)	N2—Ni—O4—C14	20.14 (15)
O1—Ni—N1—C3	−32.57 (12)	N1—Ni—O4—C14	104.43 (15)
O4—Ni—N1—C3	148.95 (12)	C18—C19—O6—C22	−7.0 (5)
N2—Ni—N1—C3	−131.33 (13)	C20—C19—O6—C22	173.0 (3)
O7—Ni—N1—C1	170.16 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7A···O4^{i}	0.791 (15)	1.984 (16)	2.769 (2)	172 (3)
O7—H7B···O1^{ii}	0.757 (15)	1.900 (16)	2.656 (2)	176 (2)
O8—H8B···O2^{ii}	0.776 (17)	2.027 (16)	2.787 (2)	166 (3)
O10—H10A···O3^{iii}	0.784 (17)	2.076 (17)	2.857 (2)	174 (3)
O10—H10B···O5^{iv}	0.796 (17)	2.12 (2)	2.851 (3)	152 (3)
C5—H5B···O9^{v}	0.97	2.49	3.430 (5)	162
C15—H15B···O9	0.97	2.52	3.451 (5)	161
O8—H8A···O10	0.832 (16)	1.913 (17)	2.731 (3)	167 (3)
C12—H12C···Cg1^{vi}	0.96	2.80	3.746 (3)	169

Symmetry codes: (i) −*x*+1/2, *y*+1/2, −*z*+1/2; (ii) −*x*+1/2, *y*−1/2, −*z*+1/2; (iii) *x*, −*y*+1, *z*−1/2; (iv) *x*, *y*+1, *z*; (v) *x*, *y*−1, *z*; (vi) −*x*, *y*−1, −*z*+1/2.

Fig. 1

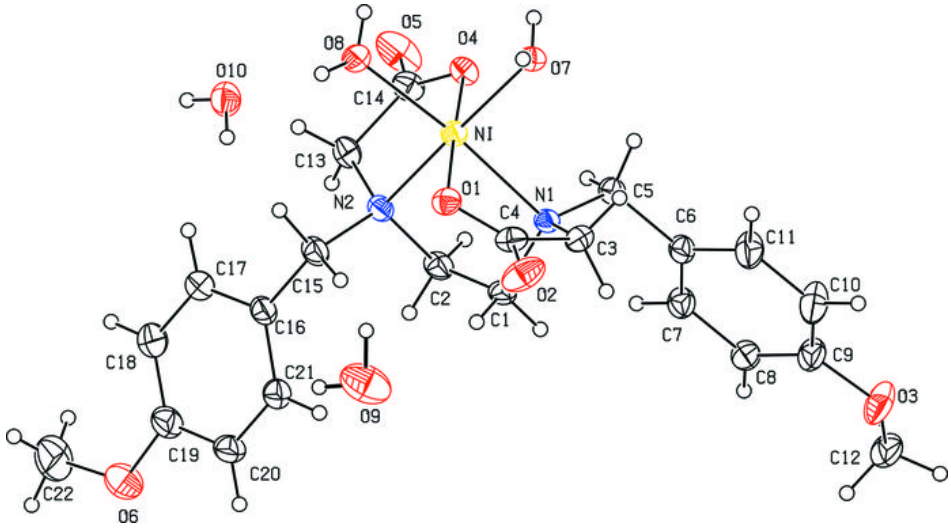
O5
O8
O4
O7
O10
C14
NI
C13
O1
N1
C5
N2
C4
C6
C11
C3
C17
C15
C2
O2
C10
C16
C1
C7
C18
C21
C9
O9
C8
O3
C19
C20
C12
C22
O6

Fig. 2