Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis{2-[tris(hydroxymethyl)methyliminomethyl]phenolato-κ^3N,O,O'}nickel(II) methanol solvate

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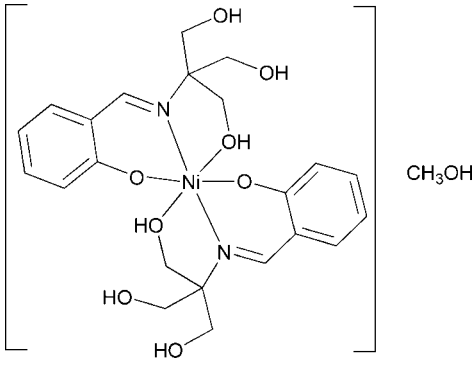
Received 31 August 2007; accepted 7 September 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.040; wR factor = 0.063; data-to-parameter ratio = 12.7.

In the crystal structure of $[Ni(C_{11}H_{14}NO_4)_2]\cdot CH_4O$, the 2-[tris(hydroxymethyl)methyliminomethyl]phenolate monoanions bind in a tridentate mode through the phenolate, alcohol and imine donor atoms to confer an octahedral geometry on nickel. Adjacent molecules are linked by O—H···O hydrogen bonds into a three-dimensional supramolecular network structure.

Related literature

See Dey *et al.* (2002) for the isostructural zinc analog. For a related Cu^{II} complex, see Ni *et al.* (2005).



Experimental

Crystal data

$[Ni(C_{11}H_{14}NO_4)_2]\cdot CH_4O$
M_r = 539.22
Orthorhombic, *Pbca*
a = 11.1900 (3) Å
b = 18.8378 (5) Å
c = 22.4202 (8) Å
V = 4726.1 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.88 mm^{-1}
T = 293 (2) K
0.1 × 0.08 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.837, T_{max} = 0.962
8161 measured reflections
4329 independent reflections
1833 reflections with $I > 2\sigma(I)$
R_{int} = 0.093

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.063
S = 0.82
4329 reflections
340 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O5^{i}	0.829 (10)	1.763 (11)	2.587 (3)	173 (4)
O7—H7B···O2^{i}	0.823 (10)	2.019 (11)	2.841 (4)	177 (3)
O8—H8A···O1^{ii}	0.824 (10)	1.795 (11)	2.616 (3)	174 (4)
O2—H2A···O3^{iii}	0.816 (10)	1.895 (12)	2.705 (3)	172 (4)
O6—H6A···O8^{iv}	0.821 (10)	1.936 (12)	2.749 (4)	171 (4)
O4—H4B···O9	0.813 (10)	2.032 (12)	2.831 (3)	167 (3)
O9—H9···O6^{v}	0.818 (10)	2.20 (2)	2.931 (3)	148 (4)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $x+\frac{1}{2}, y, -z+\frac{3}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iv) $x-\frac{1}{2}, y, -z+\frac{3}{2}$; (v) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *maXus* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2321).

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 doi:10.1107/S160053680704384X

supplementary materials

Acta Cryst. (2007). E**63**, m2537 [**doi:10.1107/S160053680704384X**]

Bis{2-[tris(hydroxymethyl)methyliminomethyl]phenolato-κ^3N,O,O'}nickel(II) methanol solvate

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Comment

Single-crystal X-ray diffraction analyses revealed that (I) is neutral, mononuclear molecule with the Ni(II) ion being octahedral coordination supplied by monoanionic and tridentate binding through the O_{phen}, O_{alk} and N_{imi} groups from two 2-[tris(hydroxymethyl)methyliminomethyl]phenolate ligands (Fig. 1). And The structure is isostructural with the zinc analog (Dey *et al.*, 2002). Selected bond lengths and bond angels are listed in Table 1. Of the three –CH2OH groups, only one is bound to the nickel center, while the other two remain free. At the same time one methanol molecule is cocrystallized in this complex which makes abundant O—H···O hydrogen bonds (Table 2) between the molecules.

Mononuclear Cu(II) complex coordinatd by one chelate 2-[tris(hydroxymethyl)methyliminomethyl]phenolate was reported in crystal structure (Ni *et al.*, 2005). And the isostructural zinc complex was reported by Dey *et al.* (2002).

Experimental

2-[tris(hydroxymethyl)methyliminomethyl]phenolate was synthesized using the salicylaldehyde and the 2-amino-2-(hydroxymethyl)propane-1,3-diol. A mixture of Nickel acetate dihydrate (0.11 g, 0.5 mmol), 2-[tris(hydroxymethyl)methyliminomethyl]phenolate (0.13 g, 0.5 mmol), and methanol (15 ml) was heated and stirring for 20 min. Suitable crystals were obtained by evaporation of the filtrate in air at room temperature.

Refinement

All H atoms besides H atoms bonded to O—H were placed at calculated positions in the riding-model approximation (C—$H_{aromatic}$ = 0.93Å and *C*—H_{methyl} = 0.96 Å), with their displacement parameters tied to those of the parent atoms by U_{iso}(H) = 1.2–1.5 times U_{eq}(C). H atoms bonded to O—H were located from different Fourier maps and were refined with restrained O—H distances with *O*—H_{methyl} = 0.82 (1) Å.

Figures

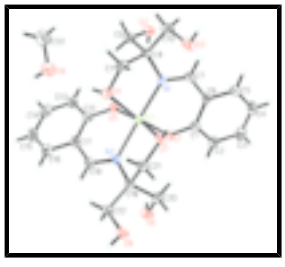


Fig. 1. View of the local coordination of Ni(II) with the coordinated atoms numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Bis{2-[tris(hydroxymethyl)methyliminomethyl]phenolato-κ3 *N*,*O*,*O*'}nickel(II) methanol solvate

Crystal data

$[Ni(C_{11}H_{14}NO_4)_2]\cdot CH_4O$	$D_x = 1.516$ Mg m^{-3}
$M_r = 539.22$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, *Pbca*	Cell parameters from 15150 reflections
$a = 11.1900$ (3) Å	$\theta = 3.4$–$27.5°$
$b = 18.8378$ (5) Å	$\mu = 0.88$ mm^{-1}
$c = 22.4202$ (8) Å	$T = 293$ (2) K
$V = 4726.1$ (2) Å^3	Block, green
$Z = 8$	0.1 × 0.08 × 0.08 mm
$F_{000} = 2272$	

Data collection

Nonius KappaCCD diffractometer	4329 independent reflections
Radiation source: fine-focus sealed tube	1833 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.093$
Detector resolution: 0.76 pixels mm^{-1}	$\theta_{max} = 25.4°$
$T = 293(2)$ K	$\theta_{min} = 3.5°$
CCD scans	$h = -13\rightarrow13$
Absorption correction: multi-scan (SORTAV; Blessing, 1995)	$k = -22\rightarrow22$
$T_{min} = 0.837$, $T_{max} = 0.962$	$l = -26\rightarrow27$
8161 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.063$	$w = 1/[\sigma^2(F_o^2) + (0.0165P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.82$	$(\Delta/\sigma)_{max} = 0.001$
4329 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
340 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
7 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > \sigma(F^2)$ 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}
Ni1	0.49786 (4)	0.214735 (17)	0.619437 (17)	0.02801 (13)
N1	0.5029 (3)	0.22644 (12)	0.53015 (10)	0.0246 (6)
N2	0.5308 (2)	0.20154 (12)	0.70767 (11)	0.0274 (8)
O7	0.6646 (2)	0.15588 (12)	0.61811 (12)	0.0351 (6)
O4	0.5970 (2)	0.31185 (12)	0.61416 (12)	0.0361 (6)
O1	0.39482 (19)	0.12901 (9)	0.61149 (10)	0.0325 (6)
O5	0.35296 (18)	0.27329 (10)	0.63661 (10)	0.0338 (6)
O3	0.6982 (2)	0.23657 (13)	0.44870 (11)	0.0442 (7)
H3A	0.744 (3)	0.2362 (17)	0.4198 (11)	0.066*
O8	0.7673 (3)	0.10518 (13)	0.79279 (10)	0.0448 (7)
H8A	0.804 (3)	0.1107 (17)	0.8242 (9)	0.067*
O2	0.3458 (2)	0.31959 (12)	0.46821 (11)	0.0396 (7)
H2A	0.301 (3)	0.2989 (16)	0.4912 (13)	0.059*
O6	0.4756 (2)	0.06072 (12)	0.76045 (12)	0.0472 (7)
H6A	0.4104 (17)	0.0748 (18)	0.7484 (17)	0.071*
O9	0.5291 (3)	0.45139 (14)	0.64657 (12)	0.0842 (10)
H9	0.518 (5)	0.468 (2)	0.6799 (9)	0.126*
C23	0.5141 (4)	0.50424 (18)	0.60590 (17)	0.0995 (18)
H23A	0.5393	0.5485	0.6229	0.149*
H23B	0.4314	0.5072	0.5949	0.149*
H23C	0.5612	0.4942	0.5711	0.149*
C1	0.3786 (3)	0.09038 (17)	0.56388 (16)	0.0277 (9)
C17	0.4001 (3)	0.30043 (17)	0.73773 (17)	0.0341 (10)
C11	0.6375 (3)	0.32327 (15)	0.55423 (16)	0.0355 (10)
H11A	0.7138	0.2997	0.5488	0.043*
H11B	0.6495	0.3737	0.5479	0.043*
C19	0.6216 (3)	0.14735 (16)	0.72315 (15)	0.0258 (9)
C16	0.3750 (4)	0.34180 (18)	0.78839 (17)	0.0491 (12)
H16A	0.4181	0.3335	0.8232	0.059*
C10	0.6118 (3)	0.29154 (16)	0.44774 (15)	0.0378 (10)
H10A	0.5540	0.2825	0.4164	0.045*
H10B	0.6504	0.3366	0.4395	0.045*
C22	0.6750 (3)	0.15574 (16)	0.78512 (14)	0.0343 (10)

H22A	0.7069	0.2033	0.7898	0.041*
H22B	0.6136	0.1486	0.8151	0.041*
C8	0.5485 (3)	0.29521 (17)	0.50759 (15)	0.0278 (9)
C18	0.4887 (3)	0.24383 (16)	0.74729 (15)	0.0367 (9)
H18A	0.5170	0.2381	0.7860	0.044*
C6	0.4161 (3)	0.11161 (17)	0.50603 (16)	0.0265 (9)
C5	0.3961 (3)	0.06634 (18)	0.45729 (15)	0.0384 (10)
H5A	0.4206	0.0809	0.4196	0.046*
C7	0.4713 (3)	0.17885 (17)	0.49257 (15)	0.0288 (10)
H7A	0.4853	0.1887	0.4525	0.035*
C21	0.7194 (3)	0.15597 (16)	0.67598 (15)	0.0347 (10)
H21A	0.7619	0.2002	0.6823	0.042*
H21B	0.7762	0.1172	0.6790	0.042*
C9	0.4411 (3)	0.34640 (16)	0.50372 (15)	0.0356 (10)
H9A	0.4116	0.3556	0.5437	0.043*
H9B	0.4679	0.3912	0.4871	0.043*
C2	0.3211 (3)	0.02409 (17)	0.56840 (17)	0.0428 (11)
H2B	0.2943	0.0088	0.6055	0.051*
C12	0.3370 (3)	0.31122 (17)	0.68496 (18)	0.0314 (10)
C20	0.5663 (3)	0.07282 (15)	0.71672 (15)	0.0342 (10)
H20A	0.5320	0.0679	0.6772	0.041*
H20B	0.6284	0.0373	0.7210	0.041*
C15	0.2898 (4)	0.39380 (18)	0.78853 (19)	0.0588 (14)
H15A	0.2742	0.4209	0.8223	0.071*
C4	0.3417 (3)	0.00142 (19)	0.4632 (2)	0.0502 (12)
H4A	0.3307	−0.0282	0.4305	0.060*
C13	0.2489 (3)	0.36394 (17)	0.68445 (16)	0.0445 (11)
H13A	0.2049	0.3720	0.6499	0.053*
C14	0.2268 (4)	0.40409 (19)	0.73480 (19)	0.0565 (14)
H14A	0.1684	0.4391	0.7332	0.068*
C3	0.3036 (4)	−0.01862 (18)	0.5195 (2)	0.0521 (13)
H3B	0.2652	−0.0620	0.5243	0.063*
H4B	0.569 (3)	0.3489 (10)	0.6263 (14)	0.071 (16)*
H7B	0.716 (2)	0.1614 (16)	0.5924 (11)	0.045 (14)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0288 (3)	0.0313 (2)	0.0239 (2)	−0.0010 (2)	−0.0014 (3)	−0.0011 (2)
N1	0.0221 (17)	0.0259 (15)	0.0259 (17)	0.0003 (16)	0.0001 (18)	−0.0009 (13)
N2	0.031 (2)	0.0269 (16)	0.0246 (18)	0.0002 (14)	−0.0028 (15)	−0.0053 (13)
O7	0.0325 (18)	0.0504 (15)	0.0224 (17)	0.0055 (13)	0.0036 (18)	−0.0002 (15)
O4	0.0416 (18)	0.0339 (15)	0.0328 (18)	−0.0023 (13)	0.0017 (16)	−0.0025 (15)
O1	0.0404 (17)	0.0362 (13)	0.0208 (15)	−0.0126 (12)	0.0043 (14)	−0.0043 (12)
O5	0.0295 (16)	0.0443 (14)	0.0276 (16)	0.0064 (12)	−0.0036 (13)	−0.0039 (13)
O3	0.036 (2)	0.0597 (16)	0.0369 (19)	0.0100 (15)	0.0150 (14)	0.0038 (15)
O8	0.043 (2)	0.0577 (15)	0.0336 (18)	0.0132 (14)	−0.0168 (15)	−0.0025 (15)
O2	0.033 (2)	0.0495 (17)	0.036 (2)	−0.0014 (14)	−0.0043 (15)	0.0075 (13)

O6	0.046 (2)	0.0514 (15)	0.0442 (18)	−0.0093 (15)	−0.0015 (18)	0.0109 (13)
O9	0.131 (3)	0.0654 (19)	0.056 (2)	0.0266 (19)	0.016 (3)	−0.0011 (16)
C23	0.156 (5)	0.073 (3)	0.069 (4)	0.020 (4)	−0.012 (4)	−0.004 (2)
C1	0.017 (2)	0.031 (2)	0.035 (3)	0.0017 (19)	0.001 (2)	0.002 (2)
C17	0.034 (3)	0.035 (2)	0.033 (3)	0.005 (2)	0.000 (2)	−0.011 (2)
C11	0.035 (3)	0.033 (2)	0.039 (3)	−0.0045 (19)	0.005 (2)	0.0059 (19)
C19	0.025 (3)	0.031 (2)	0.021 (2)	−0.0009 (19)	−0.002 (2)	−0.0037 (18)
C16	0.057 (3)	0.043 (2)	0.047 (3)	−0.001 (2)	−0.005 (2)	−0.006 (2)
C10	0.036 (3)	0.044 (2)	0.033 (3)	−0.006 (2)	0.003 (2)	0.008 (2)
C22	0.035 (3)	0.039 (2)	0.029 (3)	−0.001 (2)	−0.006 (2)	0.0011 (19)
C8	0.024 (2)	0.033 (2)	0.026 (2)	0.0019 (19)	−0.0035 (19)	0.0041 (19)
C18	0.044 (3)	0.039 (2)	0.026 (2)	−0.004 (2)	−0.007 (3)	0.0013 (18)
C6	0.026 (3)	0.029 (2)	0.025 (2)	0.0015 (18)	−0.005 (2)	−0.0056 (19)
C5	0.039 (3)	0.043 (2)	0.034 (3)	0.000 (2)	0.000 (2)	−0.001 (2)
C7	0.025 (3)	0.040 (2)	0.022 (2)	0.0035 (18)	0.0014 (19)	0.0043 (18)
C21	0.033 (3)	0.045 (2)	0.026 (3)	0.000 (2)	−0.007 (2)	0.001 (2)
C9	0.037 (3)	0.036 (2)	0.034 (3)	−0.007 (2)	−0.002 (2)	0.004 (2)
C2	0.050 (3)	0.036 (2)	0.042 (3)	−0.008 (2)	0.011 (2)	−0.003 (2)
C12	0.028 (3)	0.030 (2)	0.036 (3)	−0.0008 (19)	−0.001 (2)	0.002 (2)
C20	0.031 (3)	0.040 (2)	0.032 (3)	0.0047 (19)	0.001 (2)	0.0019 (19)
C15	0.071 (4)	0.049 (3)	0.057 (4)	0.010 (3)	0.001 (3)	−0.021 (3)
C4	0.052 (3)	0.047 (3)	0.052 (4)	0.003 (2)	−0.007 (3)	−0.020 (2)
C13	0.048 (3)	0.041 (2)	0.044 (3)	0.008 (2)	−0.001 (2)	−0.006 (2)
C14	0.054 (4)	0.041 (2)	0.074 (4)	0.012 (2)	0.008 (3)	0.001 (3)
C3	0.051 (3)	0.035 (2)	0.070 (4)	−0.013 (2)	0.002 (3)	−0.011 (2)

Geometric parameters (Å, °)

Ni1—O1	1.9922 (19)	C11—H11B	0.9700
Ni1—O5	1.998 (2)	C19—C22	1.521 (4)
Ni1—N1	2.015 (2)	C19—C21	1.531 (4)
Ni1—N2	2.028 (3)	C19—C20	1.541 (4)
Ni1—O4	2.143 (2)	C16—C15	1.367 (4)
Ni1—O7	2.170 (2)	C16—H16A	0.9300
N1—C7	1.280 (3)	C10—C8	1.519 (4)
N1—C8	1.481 (3)	C10—H10A	0.9700
N2—C18	1.283 (3)	C10—H10B	0.9700
N2—C19	1.481 (3)	C22—H22A	0.9700
O7—C21	1.435 (3)	C22—H22B	0.9700
O7—H7B	0.823 (10)	C8—C9	1.543 (4)
O4—C11	1.434 (4)	C18—H18A	0.9300
O4—H4B	0.813 (10)	C6—C5	1.404 (4)
O1—C1	1.305 (3)	C6—C7	1.441 (4)
O5—C12	1.311 (4)	C5—C4	1.373 (4)
O3—C10	1.417 (4)	C5—H5A	0.9300
O3—H3A	0.829 (10)	C7—H7A	0.9300
O8—C22	1.416 (4)	C21—H21A	0.9700
O8—H8A	0.824 (10)	C21—H21B	0.9700
O2—C9	1.424 (4)	C9—H9A	0.9700

O2—H2A	0.816 (10)	C9—H9B	0.9700
O6—C20	1.429 (4)	C2—C3	1.374 (4)
O6—H6A	0.821 (10)	C2—H2B	0.9300
O9—C23	1.361 (4)	C12—C13	1.399 (4)
O9—H9	0.818 (10)	C20—H20A	0.9700
C23—H23A	0.9600	C20—H20B	0.9700
C23—H23B	0.9600	C15—C14	1.409 (5)
C23—H23C	0.9600	C15—H15A	0.9300
C1—C2	1.408 (4)	C4—C3	1.385 (4)
C1—C6	1.421 (4)	C4—H4A	0.9300
C17—C12	1.393 (4)	C13—C14	1.381 (4)
C17—C16	1.406 (4)	C13—H13A	0.9300
C17—C18	1.471 (4)	C14—H14A	0.9300
C11—C8	1.537 (4)	C3—H3B	0.9300
C11—H11A	0.9700		
O1—Ni1—O5	89.72 (9)	C8—C10—H10B	109.7
O1—Ni1—N1	90.92 (10)	H10A—C10—H10B	108.2
O5—Ni1—N1	98.84 (10)	O8—C22—C19	109.1 (3)
O1—Ni1—N2	95.34 (9)	O8—C22—H22A	109.9
O5—Ni1—N2	91.56 (10)	C19—C22—H22A	109.9
N1—Ni1—N2	167.90 (12)	O8—C22—H22B	109.9
O1—Ni1—O4	170.66 (9)	C19—C22—H22B	109.9
O5—Ni1—O4	87.67 (9)	H22A—C22—H22B	108.3
N1—Ni1—O4	80.64 (11)	N1—C8—C10	115.0 (3)
N2—Ni1—O4	93.69 (10)	N1—C8—C11	106.9 (3)
O1—Ni1—O7	94.71 (9)	C10—C8—C11	108.4 (3)
O5—Ni1—O7	169.10 (10)	N1—C8—C9	107.3 (3)
N1—Ni1—O7	91.05 (11)	C10—C8—C9	110.0 (3)
N2—Ni1—O7	78.14 (11)	C11—C8—C9	109.1 (3)
O4—Ni1—O7	89.45 (10)	N2—C18—C17	126.6 (3)
C7—N1—C8	118.9 (3)	N2—C18—H18A	116.7
C7—N1—Ni1	124.7 (2)	C17—C18—H18A	116.7
C8—N1—Ni1	116.40 (19)	C5—C6—C1	119.5 (3)
C18—N2—C19	121.2 (3)	C5—C6—C7	116.1 (3)
C18—N2—Ni1	122.2 (2)	C1—C6—C7	124.4 (3)
C19—N2—Ni1	116.0 (2)	C4—C5—C6	122.5 (4)
C21—O7—Ni1	110.8 (2)	C4—C5—H5A	118.8
C21—O7—H7B	109 (2)	C6—C5—H5A	118.8
Ni1—O7—H7B	123 (2)	N1—C7—C6	126.6 (3)
C11—O4—Ni1	110.07 (19)	N1—C7—H7A	116.7
C11—O4—H4B	108 (2)	C6—C7—H7A	116.7
Ni1—O4—H4B	121 (2)	O7—C21—C19	108.6 (3)
C1—O1—Ni1	127.3 (2)	O7—C21—H21A	110.0
C12—O5—Ni1	124.8 (2)	C19—C21—H21A	110.0
C10—O3—H3A	115 (3)	O7—C21—H21B	110.0
C22—O8—H8A	113 (3)	C19—C21—H21B	110.0
C9—O2—H2A	106 (3)	H21A—C21—H21B	108.4
C20—O6—H6A	111 (3)	O2—C9—C8	113.2 (3)
C23—O9—H9	108 (3)	O2—C9—H9A	108.9

O9—C23—H23A	109.5	C8—C9—H9A	108.9
O9—C23—H23B	109.5	O2—C9—H9B	108.9
H23A—C23—H23B	109.5	C8—C9—H9B	108.9
O9—C23—H23C	109.5	H9A—C9—H9B	107.8
H23A—C23—H23C	109.5	C3—C2—C1	121.8 (4)
H23B—C23—H23C	109.5	C3—C2—H2B	119.1
O1—C1—C2	119.9 (3)	C1—C2—H2B	119.1
O1—C1—C6	123.3 (3)	O5—C12—C17	123.6 (3)
C2—C1—C6	116.8 (3)	O5—C12—C13	118.4 (4)
C12—C17—C16	120.3 (3)	C17—C12—C13	117.9 (3)
C12—C17—C18	124.8 (3)	O6—C20—C19	111.5 (3)
C16—C17—C18	114.7 (4)	O6—C20—H20A	109.3
O4—C11—C8	112.4 (3)	C19—C20—H20A	109.3
O4—C11—H11A	109.1	O6—C20—H20B	109.3
C8—C11—H11A	109.1	C19—C20—H20B	109.3
O4—C11—H11B	109.1	H20A—C20—H20B	108.0
C8—C11—H11B	109.1	C16—C15—C14	116.5 (4)
H11A—C11—H11B	107.9	C16—C15—H15A	121.8
N2—C19—C22	114.3 (3)	C14—C15—H15A	121.8
N2—C19—C21	104.8 (3)	C5—C4—C3	117.9 (4)
C22—C19—C21	109.8 (3)	C5—C4—H4A	121.1
N2—C19—C20	109.3 (3)	C3—C4—H4A	121.1
C22—C19—C20	109.7 (3)	C14—C13—C12	120.5 (4)
C21—C19—C20	108.6 (3)	C14—C13—H13A	119.7
C15—C16—C17	122.6 (4)	C12—C13—H13A	119.7
C15—C16—H16A	118.7	C13—C14—C15	122.3 (4)
C17—C16—H16A	118.7	C13—C14—H14A	118.9
O3—C10—C8	109.8 (3)	C15—C14—H14A	118.9
O3—C10—H10A	109.7	C2—C3—C4	121.6 (4)
C8—C10—H10A	109.7	C2—C3—H3B	119.2
O3—C10—H10B	109.7	C4—C3—H3B	119.2
O1—Ni1—N1—C7	−14.5 (3)	C20—C19—C22—O8	61.9 (3)
O5—Ni1—N1—C7	−104.4 (3)	C7—N1—C8—C10	−29.8 (4)
N2—Ni1—N1—C7	106.7 (5)	Ni1—N1—C8—C10	149.3 (2)
O4—Ni1—N1—C7	169.5 (3)	C7—N1—C8—C11	−150.2 (3)
O7—Ni1—N1—C7	80.2 (3)	Ni1—N1—C8—C11	28.9 (3)
O1—Ni1—N1—C8	166.4 (2)	C7—N1—C8—C9	92.9 (3)
O5—Ni1—N1—C8	76.6 (2)	Ni1—N1—C8—C9	−88.0 (3)
N2—Ni1—N1—C8	−72.3 (5)	O3—C10—C8—N1	−49.7 (4)
O4—Ni1—N1—C8	−9.6 (2)	O3—C10—C8—C11	69.9 (3)
O7—Ni1—N1—C8	−98.8 (2)	O3—C10—C8—C9	−170.9 (2)
O1—Ni1—N2—C18	−111.1 (3)	O4—C11—C8—N1	−40.6 (4)
O5—Ni1—N2—C18	−21.2 (3)	O4—C11—C8—C10	−165.2 (3)
N1—Ni1—N2—C18	128.1 (5)	O4—C11—C8—C9	75.1 (3)
O4—Ni1—N2—C18	66.5 (3)	C19—N2—C18—C17	179.0 (3)
O7—Ni1—N2—C18	155.2 (3)	Ni1—N2—C18—C17	8.9 (5)
O1—Ni1—N2—C19	78.3 (2)	C12—C17—C18—N2	8.3 (6)
O5—Ni1—N2—C19	168.17 (19)	C16—C17—C18—N2	−176.3 (3)
N1—Ni1—N2—C19	−42.5 (5)	O1—C1—C6—C5	179.3 (3)

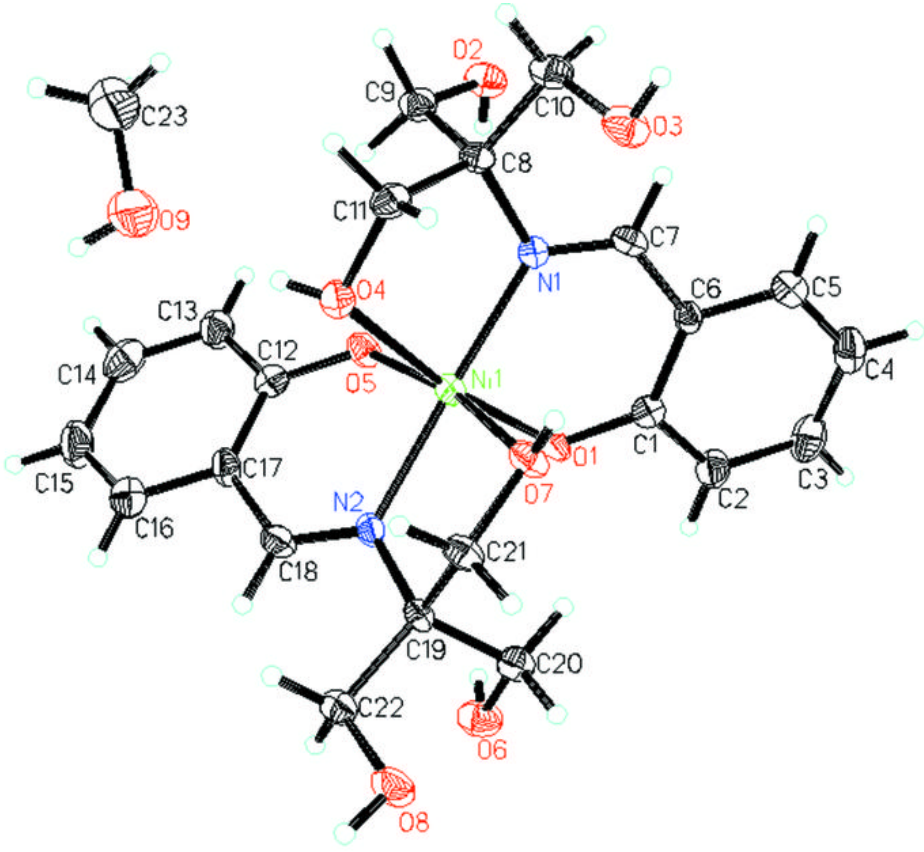
O4—Ni1—N2—C19	−104.1 (2)	C2—C1—C6—C5	−0.8 (5)
O7—Ni1—N2—C19	−15.40 (19)	O1—C1—C6—C7	−2.8 (5)
O1—Ni1—O7—C21	−108.8 (2)	C2—C1—C6—C7	177.1 (3)
O5—Ni1—O7—C21	4.9 (6)	C1—C6—C5—C4	−0.3 (5)
N1—Ni1—O7—C21	160.2 (2)	C7—C6—C5—C4	−178.4 (3)
N2—Ni1—O7—C21	−14.3 (2)	C8—N1—C7—C6	−174.6 (3)
O4—Ni1—O7—C21	79.6 (2)	Ni1—N1—C7—C6	6.4 (5)
O1—Ni1—O4—C11	−39.2 (7)	C5—C6—C7—N1	−176.3 (3)
O5—Ni1—O4—C11	−113.1 (2)	C1—C6—C7—N1	5.7 (5)
N1—Ni1—O4—C11	−13.7 (2)	Ni1—O7—C21—C19	39.6 (3)
N2—Ni1—O4—C11	155.5 (2)	N2—C19—C21—O7	−50.3 (3)
O7—Ni1—O4—C11	77.4 (2)	C22—C19—C21—O7	−173.5 (2)
O5—Ni1—O1—C1	116.5 (3)	C20—C19—C21—O7	66.4 (3)
N1—Ni1—O1—C1	17.7 (3)	N1—C8—C9—O2	−55.7 (3)
N2—Ni1—O1—C1	−151.9 (3)	C10—C8—C9—O2	70.0 (3)
O4—Ni1—O1—C1	42.8 (7)	C11—C8—C9—O2	−171.3 (3)
O7—Ni1—O1—C1	−73.4 (3)	O1—C1—C2—C3	−179.2 (3)
O1—Ni1—O5—C12	122.4 (2)	C6—C1—C2—C3	1.0 (5)
N1—Ni1—O5—C12	−146.7 (2)	Ni1—O5—C12—C17	−19.4 (4)
N2—Ni1—O5—C12	27.1 (2)	Ni1—O5—C12—C13	162.8 (2)
O4—Ni1—O5—C12	−66.6 (2)	C16—C17—C12—O5	−177.8 (3)
O7—Ni1—O5—C12	8.2 (6)	C18—C17—C12—O5	−2.7 (5)
Ni1—O1—C1—C2	168.0 (2)	C16—C17—C12—C13	−0.1 (5)
Ni1—O1—C1—C6	−12.2 (5)	C18—C17—C12—C13	175.0 (3)
Ni1—O4—C11—C8	33.7 (3)	N2—C19—C20—O6	−67.4 (3)
C18—N2—C19—C22	−10.7 (4)	C22—C19—C20—O6	58.7 (3)
Ni1—N2—C19—C22	160.0 (2)	C21—C19—C20—O6	178.8 (3)
C18—N2—C19—C21	−131.0 (3)	C17—C16—C15—C14	0.2 (6)
Ni1—N2—C19—C21	39.7 (3)	C6—C5—C4—C3	1.3 (5)
C18—N2—C19—C20	112.7 (3)	O5—C12—C13—C14	178.4 (3)
Ni1—N2—C19—C20	−76.6 (3)	C17—C12—C13—C14	0.6 (5)
C12—C17—C16—C15	−0.3 (6)	C12—C13—C14—C15	−0.7 (6)
C18—C17—C16—C15	−175.9 (3)	C16—C15—C14—C13	0.3 (6)
N2—C19—C22—O8	−174.9 (3)	C1—C2—C3—C4	0.0 (6)
C21—C19—C22—O8	−57.5 (3)	C5—C4—C3—C2	−1.2 (6)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···O5^{i}	0.829 (10)	1.763 (11)	2.587 (3)	173 (4)
O7—H7B···O2^{i}	0.823 (10)	2.019 (11)	2.841 (4)	177 (3)
O8—H8A···O1^{ii}	0.824 (10)	1.795 (11)	2.616 (3)	174 (4)
O2—H2A···O3^{iii}	0.816 (10)	1.895 (12)	2.705 (3)	172 (4)
O6—H6A···O8^{iv}	0.821 (10)	1.936 (12)	2.749 (4)	171 (4)
O4—H4B···O9	0.813 (10)	2.032 (12)	2.831 (3)	167 (3)
O9—H9···O6^{v}	0.818 (10)	2.20 (2)	2.931 (3)	148 (4)

Symmetry codes: (i) *x*+1/2, −*y*+1/2, −*z*+1; (ii) *x*+1/2, *y*, −*z*+3/2; (iii) *x*−1/2, −*y*+1/2, −*z*+1; (iv) *x*−1/2, *y*, −*z*+3/2; (v) −*x*+1, *y*+1/2, −*z*+3/2.

Fig. 1


O2
C9
C10
O3
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N1
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C13
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C14
O5
Ni1
C4
C1
O1
C17
C15
O7
C3
N2
C2
C16
C21
C18
C19
C20
C22
O6
O8