

# Bis(6-methoxy-2-[[tris(hydroxymethyl)methyl- $\kappa$ O]iminomethyl]phenolato- $\kappa^2$ N,O<sup>1</sup>)nickel(II) dihydrate

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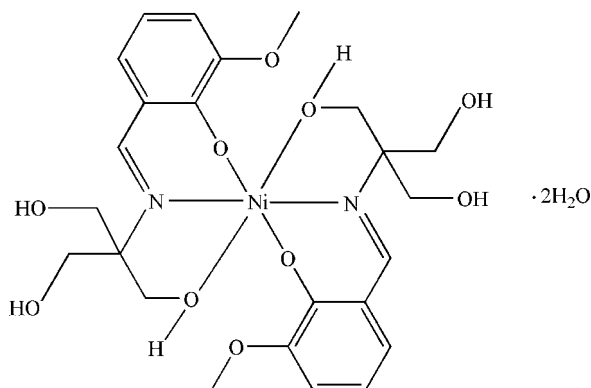
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 Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.039;  $wR$  factor = 0.117; data-to-parameter ratio = 13.1.

In the title compound,  $[\text{Ni}(\text{C}_{12}\text{H}_{16}\text{NO}_5)_2] \cdot 2\text{H}_2\text{O}$ , the  $\text{Ni}^{\text{II}}$  atom is coordinated by four O atoms and two N atoms from the two 6-methoxy-2-[[tris(hydroxymethyl)methyl]iminomethyl]phenolato ligands in a distorted octahedral coordination geometry. O—H...O hydrogen bonds link the complexes and uncoordinated water molecules into two-dimensional networks parallel to (001).

## Related literature

For the applications of Schiff-base complexes, see: Kritagawa & Kondo (1998); Zhang *et al.* (1998); Yaghi *et al.* (1996).



## Experimental

### Crystal data

$[\text{Ni}(\text{C}_{12}\text{H}_{16}\text{NO}_5)_2] \cdot 2\text{H}_2\text{O}$   
 $M_r = 603.26$   
 Monoclinic,  $P2_1/c$

$a = 12.0142$  (10) Å  
 $b = 10.9876$  (10) Å  
 $c = 20.324$  (2) Å

$\beta = 97.501$  (1)°  
 $V = 2660.0$  (4) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 0.80$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.44 \times 0.29 \times 0.20$  mm

### Data collection

Bruker APEXII CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)  
 $T_{\text{min}} = 0.721$ ,  $T_{\text{max}} = 0.857$

13321 measured reflections  
 4933 independent reflections  
 4436 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.043$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.117$   
 $S = 1.00$   
 4933 reflections  
 376 parameters  
 8 restraints

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.44$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1...O2 <sup>i</sup>	0.82	1.85	2.670 (3)	179
O2—H2A...O11 <sup>ii</sup>	0.82	1.91	2.666 (3)	152
O2—H2A...O12 <sup>ii</sup>	0.82	2.37	3.010 (3)	135
O5—H5...O6 <sup>iii</sup>	0.82	1.87	2.691 (3)	174
O6—H6...O3 <sup>iv</sup>	0.82	1.89	2.671 (2)	159
O10—H10A...O5 <sup>v</sup>	0.82 (3)	1.93 (3)	2.751 (3)	175 (5)
O8—H1AA...O7 <sup>v</sup>	0.82 (2)	1.972 (11)	2.775 (4)	166 (4)
O4—H4AA...O8 <sup>v</sup>	0.82 (3)	1.88 (4)	2.686 (3)	170 (4)
O8—H1BB...O2 <sup>vi</sup>	0.82 (3)	2.16 (3)	2.962 (3)	167 (4)
O7—H2BB...O9 <sup>vi</sup>	0.82 (2)	2.055 (10)	2.862 (4)	168 (4)
O7—H2AA...O1	0.81 (3)	1.84 (3)	2.641 (3)	169 (4)

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2378).

## References

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**supplementary materials**

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## Bis(6-methoxy-2-[[tris(hydroxymethyl)methyl- $\kappa$ O]iminomethyl]phenolato- $\kappa^2N,O^1$ )nickel(II) dihydrate

T. Zhou, R.-J. Zhou and Z. An

### Comment

Polymeric metal complexes containing Schiff-base ligands are of interest because of their useful chemical or physical properties (Zhang *et al.*, 1998; Kritagawa & Kondo, 1998; Yaghi *et al.*, 1996). Herein, we report a new crystal structure containing the Schiff-base ligand 6-methoxy-2-[[tris(hydroxymethyl)methyl]iminomethyl]phenol (denoted HL).

As shown in Figure 1, the asymmetric unit of the complex comprises two  $L^-$  ligands, one  $Ni^{II}$  atom and two lattice water molecules. The  $Ni^{II}$  atom is hexa-coordinated by four O atoms and two N atoms from the two  $L^-$  ligands, giving a distorted octahedral coordination geometry. The Ni—O and Ni—N bond distances are within normal ranges. The  $[NiL_2]$  complexes form an extensive network of O—H $\cdots$ O interactions involving the lattice water molecules, giving 2-D networks parallel to the (001) planes (Fig. 2).

### Experimental

The complex was synthesized by refluxing HL (0.050 g, 0.2 mmol) and  $NiCl_2 \cdot 6H_2O$  (0.048 g, 0.2 mmol) in the mixed solution ( $CH_3OH:H_2O = 4:1$ ) until all solid was dissolved. The solution was then cooled to room temperature and filtered. Green crystals for X-ray diffraction analysis were obtained by slow evaporation of the filtrate. Elemental analysis calculated: C 47.74, H 5.97, N 4.64 %; found: C 47.69, H 5.51, N 4.58 %.

### Refinement

All H atoms bound to C were placed geometrically with C—H = 0.93 (aromatic H), 0.96 (methyl H) or 0.97 Å (methylene H) and refined as riding with  $U_{iso}(H) = 1.2U_{eq}(C)$  (aromatic and methylene H) or  $1.5U_{eq}(C)$  (methyl H). The H atoms of the water molecule were located from difference density maps and refined with distance restraints of  $d(H\cdots H) = 1.38(2)$  Å,  $d(O—H) = 0.82(1)$  Å. The H atoms of the hydroxyl groups were placed geometrically with O—H = 0.82 Å.

### Figures

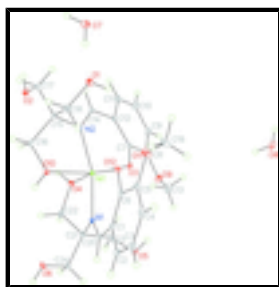


Fig. 1. Molecular structure with displacement ellipsoids drawn at the 30% probability level for non-H atoms.

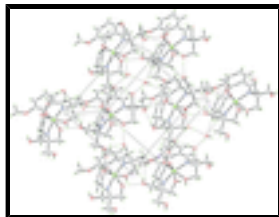


Fig. 2. Packing diagram viewed approximately along the *c* axis, showing the complex network of O—H...O hydrogen bonds (dashed lines).

## Bis(6-methoxy-2-[[tris(hydroxymethyl)methyl-κO]iminomethyl]phenolato-κ<sup>2</sup>N,O<sup>1</sup>)nickel(II) dihydrate

### Crystal data

[Ni(C<sub>12</sub>H<sub>16</sub>N<sub>1</sub>O<sub>5</sub>)<sub>2</sub>]·2H<sub>2</sub>O

*M<sub>r</sub>* = 603.26

Monoclinic, *P*2<sub>1</sub>/*c*

Hall symbol: -*P* 2ybc

*a* = 12.0142 (10) Å

*b* = 10.9876 (10) Å

*c* = 20.324 (2) Å

β = 97.501 (1)°

*V* = 2660.0 (4) Å<sup>3</sup>

*Z* = 4

*F*<sub>000</sub> = 1272

*D<sub>x</sub>* = 1.506 Mg m<sup>-3</sup>

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 4933 reflections

θ = 2.0–25.5°

μ = 0.80 mm<sup>-1</sup>

*T* = 293 K

Block, green

0.44 × 0.29 × 0.20 mm

### Data collection

Bruker APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

*T* = 293 K

φ and ω scans

Absorption correction: multi-scan  
(SADABS; Sheldrick, 2003)

*T*<sub>min</sub> = 0.721, *T*<sub>max</sub> = 0.857

13321 measured reflections

4933 independent reflections

4436 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.043

θ<sub>max</sub> = 25.5°

θ<sub>min</sub> = 2.0°

*h* = -14→11

*k* = -13→13

*l* = -24→21

### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.039

*wR*(*F*<sup>2</sup>) = 0.117

*S* = 1.00

4933 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.069P)^2 + 2.387P]$$

where *P* = (*F*<sub>o</sub><sup>2</sup> + 2*F*<sub>c</sub><sup>2</sup>)/3

(Δ/σ)<sub>max</sub> = 0.032

Δρ<sub>max</sub> = 0.38 e Å<sup>-3</sup>

376 parameters

$$\Delta\rho_{\min} = -0.44 \text{ e } \text{\AA}^{-3}$$

8 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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.9480 (2)	0.2452 (2)	0.86933 (11)	0.0233 (5)
C2	1.0283 (2)	0.2677 (3)	0.92480 (12)	0.0311 (6)
H2	1.1023	0.2433	0.9242	0.037*
C3	0.9989 (3)	0.3243 (3)	0.97874 (13)	0.0386 (7)
H3	1.0524	0.3390	1.0153	0.046*
C4	0.8871 (3)	0.3610 (3)	0.97952 (14)	0.0391 (7)
H4	0.8671	0.4003	1.0168	0.047*
C5	0.8073 (2)	0.3402 (2)	0.92655 (13)	0.0307 (6)
C6	0.8335 (2)	0.2797 (2)	0.86879 (11)	0.0228 (5)
C7	0.6684 (2)	0.3019 (2)	0.62935 (12)	0.0231 (5)
C8	0.6897 (3)	0.3685 (2)	0.57276 (13)	0.0325 (6)
C9	0.6097 (3)	0.3783 (3)	0.51866 (15)	0.0468 (8)
H9	0.6251	0.4238	0.4823	0.056*
C10	0.5051 (3)	0.3210 (3)	0.51678 (16)	0.0515 (9)
H10	0.4509	0.3307	0.4801	0.062*
C11	0.4834 (3)	0.2522 (3)	0.56833 (15)	0.0404 (7)
H11	0.4143	0.2136	0.5668	0.048*
C12	0.5639 (2)	0.2379 (2)	0.62428 (12)	0.0269 (5)
C13	0.8357 (4)	0.4634 (4)	0.52069 (18)	0.0638 (11)
H13A	0.8300	0.3993	0.4883	0.096*
H13B	0.9128	0.4872	0.5312	0.096*
H13C	0.7921	0.5319	0.5030	0.096*
C14	0.5356 (2)	0.1521 (2)	0.67266 (13)	0.0265 (5)
H14	0.4621	0.1238	0.6679	0.032*
C15	0.5631 (2)	0.0166 (2)	0.76457 (13)	0.0264 (5)
C16	0.6607 (2)	-0.0731 (2)	0.77949 (14)	0.0312 (6)
H16A	0.6454	-0.1291	0.8141	0.037*
H16B	0.6694	-0.1199	0.7400	0.037*
C17	0.4562 (2)	-0.0513 (3)	0.73736 (15)	0.0344 (6)

## supplementary materials

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H17A	0.4381	-0.1104	0.7698	0.041*
H17B	0.3943	0.0059	0.7296	0.041*
C18	0.5411 (3)	0.0770 (3)	0.82811 (14)	0.0370 (6)
H18A	0.5226	0.0156	0.8592	0.044*
H18B	0.6083	0.1190	0.8478	0.044*
C19	0.6632 (3)	0.4399 (3)	0.97696 (15)	0.0488 (8)
H19A	0.6755	0.3908	1.0163	0.073*
H19B	0.5850	0.4603	0.9678	0.073*
H19C	0.7068	0.5131	0.9835	0.073*
C20	0.9938 (2)	0.1930 (2)	0.81394 (12)	0.0228 (5)
H20	1.0712	0.1823	0.8182	0.027*
C21	0.9989 (2)	0.1161 (2)	0.70684 (12)	0.0229 (5)
C22	0.9245 (2)	0.0216 (2)	0.66780 (12)	0.0260 (5)
H22A	0.9502	0.0080	0.6251	0.031*
H22B	0.9290	-0.0549	0.6918	0.031*
C23	1.0163 (2)	0.2240 (2)	0.66147 (12)	0.0276 (5)
H23A	1.0622	0.1977	0.6282	0.033*
H23B	0.9441	0.2490	0.6386	0.033*
C24	1.1143 (2)	0.0572 (2)	0.72948 (13)	0.0282 (5)
H24A	1.1428	0.0193	0.6919	0.034*
H24B	1.1675	0.1189	0.7475	0.034*
N1	0.93758 (17)	0.16035 (17)	0.75960 (9)	0.0199 (4)
N2	0.60221 (17)	0.11097 (18)	0.72161 (10)	0.0228 (4)
Ni1	0.76754 (2)	0.15968 (3)	0.742167 (14)	0.01963 (12)
O1	0.4508 (2)	0.1618 (2)	0.81592 (14)	0.0531 (6)
H1	0.4759	0.2313	0.8178	0.080*
O2	0.46970 (17)	-0.11101 (19)	0.67775 (11)	0.0418 (5)
H2A	0.4079	-0.1255	0.6571	0.063*
O3	0.74281 (14)	0.30277 (15)	0.68125 (8)	0.0233 (4)
O4	0.81045 (15)	0.06375 (17)	0.65815 (9)	0.0301 (4)
O5	1.06823 (16)	0.32512 (17)	0.69579 (10)	0.0341 (4)
H5	1.0200	0.3734	0.7042	0.051*
O6	1.10157 (16)	-0.03050 (19)	0.77806 (11)	0.0399 (5)
H6	1.1596	-0.0702	0.7859	0.060*
O7	0.2701 (2)	0.1557 (2)	0.87725 (16)	0.0584 (7)
O8	0.6646 (2)	0.8964 (2)	0.60240 (12)	0.0524 (6)
O9	0.79479 (19)	0.4219 (2)	0.57859 (10)	0.0440 (5)
O10	0.76155 (15)	-0.00657 (17)	0.80057 (10)	0.0311 (4)
O11	0.69602 (18)	0.3743 (2)	0.92284 (10)	0.0446 (5)
O12	0.75320 (14)	0.26451 (16)	0.82089 (8)	0.0258 (4)
H10A	0.815 (2)	-0.054 (3)	0.803 (2)	0.080*
H1AA	0.690 (3)	0.8269 (14)	0.603 (2)	0.080*
H2AA	0.329 (2)	0.151 (3)	0.862 (2)	0.080*
H4AA	0.768 (3)	0.008 (3)	0.645 (2)	0.080*
H1BB	0.605 (2)	0.902 (3)	0.618 (2)	0.080*
H2BB	0.243 (3)	0.0900 (16)	0.886 (2)	0.080*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0281 (12)	0.0218 (12)	0.0198 (11)	-0.0011 (10)	0.0026 (9)	-0.0003 (9)
C2	0.0308 (13)	0.0363 (15)	0.0247 (13)	0.0002 (12)	-0.0020 (10)	0.0005 (11)
C3	0.0432 (17)	0.0480 (18)	0.0220 (13)	-0.0017 (14)	-0.0059 (12)	-0.0053 (12)
C4	0.0487 (18)	0.0460 (17)	0.0222 (13)	0.0024 (14)	0.0036 (12)	-0.0104 (12)
C5	0.0367 (15)	0.0310 (14)	0.0247 (13)	0.0056 (11)	0.0052 (11)	-0.0026 (10)
C6	0.0300 (13)	0.0203 (11)	0.0177 (11)	-0.0016 (10)	0.0022 (9)	0.0016 (9)
C7	0.0293 (13)	0.0164 (11)	0.0232 (12)	0.0003 (10)	0.0022 (10)	-0.0012 (9)
C8	0.0461 (16)	0.0242 (13)	0.0265 (13)	-0.0038 (12)	0.0026 (11)	0.0015 (10)
C9	0.073 (2)	0.0381 (16)	0.0257 (14)	-0.0042 (16)	-0.0061 (14)	0.0096 (12)
C10	0.065 (2)	0.0432 (18)	0.0374 (17)	-0.0052 (16)	-0.0254 (16)	0.0094 (14)
C11	0.0407 (16)	0.0311 (15)	0.0442 (17)	-0.0031 (12)	-0.0144 (13)	0.0020 (12)
C12	0.0294 (13)	0.0217 (12)	0.0276 (12)	0.0018 (10)	-0.0035 (10)	-0.0005 (10)
C13	0.094 (3)	0.053 (2)	0.052 (2)	-0.018 (2)	0.040 (2)	-0.0003 (17)
C14	0.0231 (12)	0.0206 (12)	0.0346 (14)	-0.0017 (10)	-0.0015 (10)	-0.0026 (10)
C15	0.0262 (12)	0.0206 (12)	0.0333 (13)	-0.0046 (10)	0.0068 (10)	0.0036 (10)
C16	0.0318 (14)	0.0220 (13)	0.0391 (14)	-0.0031 (11)	0.0022 (11)	0.0055 (11)
C17	0.0268 (13)	0.0267 (14)	0.0494 (17)	-0.0059 (11)	0.0037 (12)	0.0054 (12)
C18	0.0429 (16)	0.0336 (15)	0.0375 (15)	-0.0038 (13)	0.0170 (12)	0.0044 (12)
C19	0.056 (2)	0.057 (2)	0.0362 (16)	0.0188 (16)	0.0167 (14)	-0.0120 (15)
C20	0.0223 (12)	0.0202 (11)	0.0253 (12)	-0.0011 (10)	0.0011 (9)	0.0006 (9)
C21	0.0248 (12)	0.0220 (12)	0.0225 (11)	0.0003 (10)	0.0056 (9)	-0.0032 (9)
C22	0.0307 (13)	0.0203 (12)	0.0265 (12)	0.0007 (10)	0.0022 (10)	-0.0055 (10)
C23	0.0331 (13)	0.0269 (13)	0.0239 (12)	-0.0017 (11)	0.0080 (10)	-0.0004 (10)
C24	0.0267 (13)	0.0261 (13)	0.0322 (13)	0.0031 (10)	0.0054 (10)	-0.0007 (10)
N1	0.0236 (10)	0.0174 (10)	0.0190 (10)	0.0009 (8)	0.0044 (8)	0.0006 (7)
N2	0.0221 (10)	0.0186 (10)	0.0276 (11)	-0.0009 (8)	0.0028 (8)	-0.0005 (8)
Ni1	0.02003 (18)	0.01864 (18)	0.01981 (18)	-0.00071 (11)	0.00109 (12)	-0.00067 (11)
O1	0.0484 (14)	0.0359 (12)	0.0817 (18)	0.0007 (10)	0.0338 (13)	-0.0071 (12)
O2	0.0364 (11)	0.0328 (11)	0.0528 (13)	-0.0080 (9)	-0.0074 (9)	-0.0070 (10)
O3	0.0269 (9)	0.0198 (8)	0.0223 (8)	-0.0042 (7)	-0.0004 (7)	0.0010 (7)
O4	0.0286 (9)	0.0285 (10)	0.0319 (10)	-0.0018 (8)	-0.0014 (7)	-0.0101 (8)
O5	0.0351 (11)	0.0263 (10)	0.0421 (11)	-0.0068 (8)	0.0096 (9)	-0.0016 (8)
O6	0.0309 (10)	0.0326 (11)	0.0560 (13)	0.0111 (9)	0.0046 (9)	0.0139 (9)
O7	0.0449 (14)	0.0510 (15)	0.0836 (19)	0.0014 (11)	0.0248 (13)	-0.0003 (13)
O8	0.0615 (16)	0.0469 (14)	0.0485 (13)	-0.0191 (12)	0.0057 (11)	-0.0043 (11)
O9	0.0523 (13)	0.0463 (12)	0.0351 (11)	-0.0156 (10)	0.0125 (9)	0.0085 (9)
O10	0.0281 (9)	0.0250 (9)	0.0390 (10)	0.0007 (8)	-0.0004 (8)	0.0041 (8)
O11	0.0417 (12)	0.0646 (14)	0.0275 (10)	0.0182 (11)	0.0046 (9)	-0.0162 (10)
O12	0.0246 (9)	0.0298 (9)	0.0222 (8)	0.0023 (7)	0.0008 (7)	-0.0059 (7)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

C1—C2	1.406 (3)	C17—H17B	0.970
C1—C6	1.426 (4)	C18—O1	1.427 (4)
C1—C20	1.435 (3)	C18—H18A	0.970

## supplementary materials

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C2—C3	1.347 (4)	C18—H18B	0.970
C2—H2	0.930	C19—O11	1.414 (3)
C3—C4	1.405 (5)	C19—H19A	0.960
C3—H3	0.930	C19—H19B	0.960
C4—C5	1.364 (4)	C19—H19C	0.960
C4—H4	0.930	C20—N1	1.269 (3)
C5—O11	1.381 (3)	C20—H20	0.930
C5—C6	1.419 (3)	C21—N1	1.461 (3)
C6—O12	1.289 (3)	C21—C22	1.524 (3)
C7—O3	1.290 (3)	C21—C23	1.533 (3)
C7—C8	1.414 (4)	C21—C24	1.545 (3)
C7—C12	1.431 (4)	C22—O4	1.435 (3)
C8—C9	1.366 (4)	C22—H22A	0.970
C8—O9	1.384 (4)	C22—H22B	0.970
C9—C10	1.402 (5)	C23—O5	1.413 (3)
C9—H9	0.930	C23—H23A	0.970
C10—C11	1.345 (5)	C23—H23B	0.970
C10—H10	0.930	C24—O6	1.402 (3)
C11—C12	1.402 (4)	C24—H24A	0.970
C11—H11	0.930	C24—H24B	0.970
C12—C14	1.435 (4)	N1—Ni1	2.027 (2)
C13—O9	1.409 (4)	N2—Ni1	2.047 (2)
C13—H13A	0.960	Ni1—O12	1.9971 (17)
C13—H13B	0.960	Ni1—O3	1.9993 (17)
C13—H13C	0.960	Ni1—O4	2.1266 (18)
C14—N2	1.275 (3)	Ni1—O10	2.1847 (19)
C14—H14	0.930	O1—H1	0.820
C15—N2	1.471 (3)	O2—H2A	0.820
C15—C18	1.506 (4)	O4—H4AA	0.82 (3)
C15—C17	1.526 (3)	O5—H5	0.820
C15—C16	1.531 (4)	O6—H6	0.820
C16—O10	1.432 (3)	O7—H2AA	0.81 (3)
C16—H16A	0.970	O7—H2BB	0.82 (2)
C16—H16B	0.970	O8—H1AA	0.82 (2)
C17—O2	1.406 (4)	O8—H1BB	0.82 (3)
C17—H17A	0.970	O10—H10A	0.82 (3)
C2—C1—C6	121.3 (2)	O11—C19—H19B	109.5
C2—C1—C20	114.0 (2)	H19A—C19—H19B	109.5
C6—C1—C20	124.6 (2)	O11—C19—H19C	109.5
C3—C2—C1	120.6 (3)	H19A—C19—H19C	109.5
C3—C2—H2	119.7	H19B—C19—H19C	109.5
C1—C2—H2	119.7	N1—C20—C1	125.5 (2)
C2—C3—C4	119.6 (3)	N1—C20—H20	117.2
C2—C3—H3	120.2	C1—C20—H20	117.2
C4—C3—H3	120.2	N1—C21—C22	106.9 (2)
C5—C4—C3	121.2 (3)	N1—C21—C23	107.81 (19)
C5—C4—H4	119.4	C22—C21—C23	109.3 (2)
C3—C4—H4	119.4	N1—C21—C24	116.1 (2)
C4—C5—O11	125.0 (2)	C22—C21—C24	108.2 (2)

C4—C5—C6	121.5 (3)	C23—C21—C24	108.4 (2)
O11—C5—C6	113.5 (2)	O4—C22—C21	109.65 (19)
O12—C6—C5	117.4 (2)	O4—C22—H22A	109.7
O12—C6—C1	126.7 (2)	C21—C22—H22A	109.7
C5—C6—C1	115.9 (2)	O4—C22—H22B	109.7
O3—C7—C8	118.8 (2)	C21—C22—H22B	109.7
O3—C7—C12	124.8 (2)	H22A—C22—H22B	108.2
C8—C7—C12	116.4 (2)	O5—C23—C21	113.4 (2)
C9—C8—O9	125.0 (3)	O5—C23—H23A	108.9
C9—C8—C7	120.8 (3)	C21—C23—H23A	108.9
O9—C8—C7	114.2 (2)	O5—C23—H23B	108.9
C8—C9—C10	121.3 (3)	C21—C23—H23B	108.9
C8—C9—H9	119.3	H23A—C23—H23B	107.7
C10—C9—H9	119.3	O6—C24—C21	108.7 (2)
C11—C10—C9	119.7 (3)	O6—C24—H24A	109.9
C11—C10—H10	120.2	C21—C24—H24A	109.9
C9—C10—H10	120.2	O6—C24—H24B	109.9
C10—C11—C12	120.8 (3)	C21—C24—H24B	109.9
C10—C11—H11	119.6	H24A—C24—H24B	108.3
C12—C11—H11	119.6	C20—N1—C21	118.1 (2)
C11—C12—C7	120.6 (2)	C20—N1—Ni1	124.24 (17)
C11—C12—C14	115.5 (2)	C21—N1—Ni1	117.62 (15)
C7—C12—C14	123.9 (2)	C14—N2—C15	119.8 (2)
O9—C13—H13A	109.5	C14—N2—Ni1	124.04 (18)
O9—C13—H13B	109.5	C15—N2—Ni1	116.08 (15)
H13A—C13—H13B	109.5	O12—Ni1—O3	91.21 (7)
O9—C13—H13C	109.5	O12—Ni1—N1	92.77 (7)
H13A—C13—H13C	109.5	O3—Ni1—N1	99.80 (7)
H13B—C13—H13C	109.5	O12—Ni1—N2	97.55 (8)
N2—C14—C12	126.0 (2)	O3—Ni1—N2	91.00 (7)
N2—C14—H14	117.0	N1—Ni1—N2	164.91 (8)
C12—C14—H14	117.0	O12—Ni1—O4	169.89 (7)
N2—C15—C18	107.7 (2)	O3—Ni1—O4	85.67 (7)
N2—C15—C17	116.6 (2)	N1—Ni1—O4	78.33 (7)
C18—C15—C17	107.0 (2)	N2—Ni1—O4	92.12 (8)
N2—C15—C16	106.0 (2)	O12—Ni1—O10	91.98 (7)
C18—C15—C16	109.2 (2)	O3—Ni1—O10	168.87 (7)
C17—C15—C16	110.1 (2)	N1—Ni1—O10	90.69 (7)
O10—C16—C15	109.1 (2)	N2—Ni1—O10	78.01 (7)
O10—C16—H16A	109.9	O4—Ni1—O10	92.89 (7)
C15—C16—H16A	109.9	C18—O1—H1	109.5
O10—C16—H16B	109.9	C17—O2—H2A	109.5
C15—C16—H16B	109.9	C7—O3—Ni1	122.09 (15)
H16A—C16—H16B	108.3	C22—O4—Ni1	112.16 (13)
O2—C17—C15	110.7 (2)	C22—O4—H4AA	110 (3)
O2—C17—H17A	109.5	Ni1—O4—H4AA	115 (3)
C15—C17—H17A	109.5	C23—O5—H5	109.5
O2—C17—H17B	109.5	C24—O6—H6	109.5
C15—C17—H17B	109.5	H2AA—O7—H2BB	115 (3)

## supplementary materials

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H17A—C17—H17B	108.1	H1AA—O8—H1BB	114 (3)
O1—C18—C15	110.7 (2)	C8—O9—C13	118.7 (3)
O1—C18—H18A	109.5	C16—O10—Ni1	110.53 (14)
C15—C18—H18A	109.5	C16—O10—H10A	109 (3)
O1—C18—H18B	109.5	Ni1—O10—H10A	118 (3)
C15—C18—H18B	109.5	C5—O11—C19	117.5 (2)
H18A—C18—H18B	108.1	C6—O12—Ni1	123.07 (16)
O11—C19—H19A	109.5		

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ O2 <sup>i</sup>	0.82	1.85	2.670 (3)	179
O2—H2A $\cdots$ O11 <sup>ii</sup>	0.82	1.91	2.666 (3)	152
O2—H2A $\cdots$ O12 <sup>ii</sup>	0.82	2.37	3.010 (3)	135
O5—H5 $\cdots$ O6 <sup>iii</sup>	0.82	1.87	2.691 (3)	174
O6—H6 $\cdots$ O3 <sup>iv</sup>	0.82	1.89	2.671 (2)	159
O10—H10A $\cdots$ O5 <sup>iv</sup>	0.82 (3)	1.93 (3)	2.751 (3)	175 (5)
O8—H1AA $\cdots$ O7 <sup>i</sup>	0.82 (2)	1.972 (11)	2.775 (4)	166 (4)
O4—H4AA $\cdots$ O8 <sup>v</sup>	0.82 (3)	1.88 (4)	2.686 (3)	170 (4)
O8—H1BB $\cdots$ O2 <sup>vi</sup>	0.82 (3)	2.16 (3)	2.962 (3)	167 (4)
O7—H2BB $\cdots$ O9 <sup>ii</sup>	0.82 (2)	2.055 (10)	2.862 (4)	168 (4)
O7—H2AA $\cdots$ O1	0.81 (3)	1.84 (3)	2.641 (3)	169 (4)

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

Fig. 1

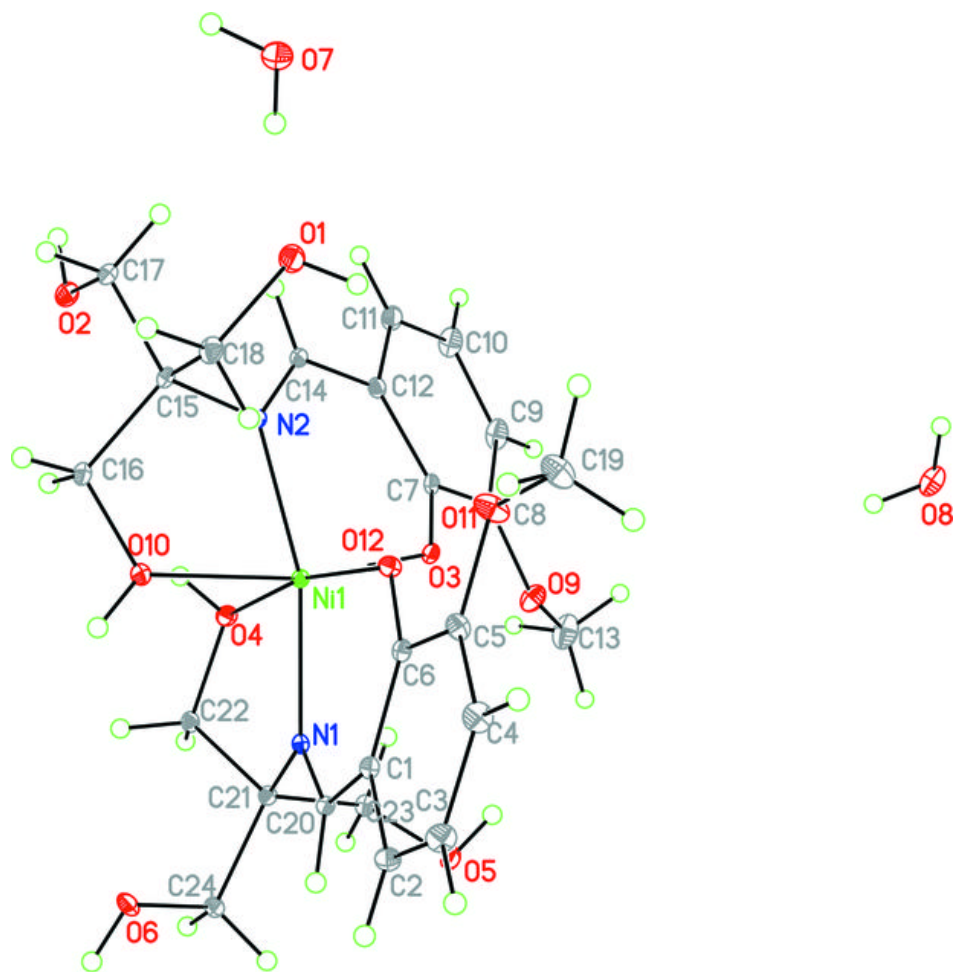


Fig. 2

