

(Di-2-pyridylamine- κ^2N^1, N^1')bis-(methacrylate- κO)nickel(II) sesquihydrate

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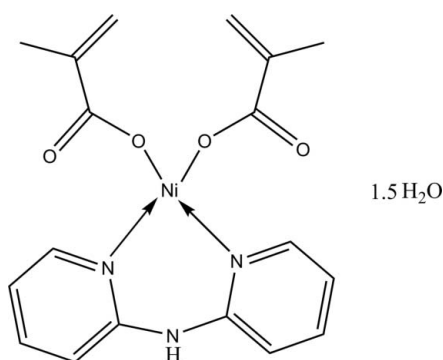
Received 25 February 2008; accepted 3 April 2008

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(C-C) = 0.005$ Å; disorder in solvent or counterion; R factor = 0.036; wR factor = 0.085; data-to-parameter ratio = 11.3.

In the title mononuclear complex, $[Ni(C_4H_5O_2)_2(C_{10}H_9N_3)] \cdot 1.5H_2O$, the Ni^{II} ion is in a distorted square-planar coordination environment, formed by two O atoms from two methacrylate ligands and two N atoms from a bis-chelating dipyridylamine ligand. In the crystal structure, intermolecular $O-H \cdots O$ and $N-H \cdots O$ hydrogen bonds link complex molecules and water molecules into one-dimensional chains.

Related literature

For the Cu analog of the title compound, see: Liu, *et al.* (2006). For related literature, see: Carabias-Martínez *et al.* (2006); Matsui *et al.* (1997); Wang *et al.* (1997); Wu *et al.* (2002).



Experimental

Crystal data

$[Ni(C_4H_5O_2)_2(C_{10}H_9N_3)] \cdot 1.5H_2O$ $c = 15.5396$ (17) Å
 $M_r = 427.10$ $\beta = 101.483$ (2)°
 Monoclinic, $P2_1/n$ $V = 2003.8$ (4) Å³
 $a = 8.3686$ (9) Å $Z = 4$
 $b = 15.7235$ (16) Å Mo $K\alpha$ radiation

$\mu = 1.00$ mm⁻¹
 $T = 293$ (2) K

0.29 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer 9960 measured reflections
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996) 3512 independent reflections
 $T_{min} = 0.740$, $T_{max} = 0.872$ 2720 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.077$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.084$ $\Delta\rho_{max} = 0.32$ e Å⁻³
 $S = 1.00$ $\Delta\rho_{min} = -0.24$ e Å⁻³
 3512 reflections
 311 parameters
 13 restraints

Table 1

Selected geometric parameters (Å, °).

Ni1—O3	1.9669 (18)	Ni1—N1	1.9801 (18)
Ni1—N2	1.980 (2)	Ni1—O1	1.9843 (17)
O3—Ni1—N2	93.16 (8)	O3—Ni1—O1	92.24 (8)
O3—Ni1—N1	154.32 (8)	N2—Ni1—O1	153.74 (8)
N2—Ni1—N1	92.47 (8)	N1—Ni1—O1	93.70 (7)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O6—H6B ⁱ ···O3 ⁱ	0.85	2.31	3.030	143
N3—H19···O5 ⁱⁱ	0.86	1.99	2.837 (3)	170
O5—H20···O2	0.96 (3)	1.75 (3)	2.698 (3)	169 (2)
O5—H21···O4 ⁱⁱⁱ	0.92 (3)	1.88 (3)	2.789 (3)	169 (3)

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

Data collection: *SMART* (Bruker, 1996); cell refinement: *SAINT* (Bruker, 1996); data reduction: *SAINT*; 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*.

The authors thank the Postgraduate Foundation of Daqing Normal University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2600).

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supplementary materials

Acta Cryst. (2008). E64, m640 [doi:10.1107/S1600536808009008]

(Di-2-pyridylamine- κ^2N^1,N^1')bis(methacrylato- κO)nickel(II) sesquihydrate

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Comment

Methacrylic acid and its derivatives are biologically active compounds which are widely used as herbicides and plantgrowth substances (Matsui *et al.*, 1997; Carabias-Martínez *et al.*, 2006). Due to their versatile bonding modes with metal ions, they have also been used in the synthesis of mononuclear or multi-nuclear compounds (Wang *et al.*, 1997; Wu *et al.*, 2002). In order to develop some new topological structures, the reaction system of a nickel(II)chloride with methacrylic acid and dipyridin-2-ylamine has been explored.

Herein we report the structure of the title compound (Fig. 1). It is isostructural with the Cu analog (Liu *et al.*, 2006) but we have located and refined an additional half of a water solvent molecule. The Ni^{II} ion is in a distorted square-planar coordination environment, formed by two O atoms from two methacrylate ligands and two N atoms from a bis-chelating dipyridin-2-ylamine ligand. In the crystal structure, intermolecular O—H \cdots O and N—H \cdots O hydrogen bonds link complex molecules and water molecules into a to form one-dimensional chains (Fig. 2).

Experimental

Methacrylic acid and dipyridin-2-ylamine are commercially available, and they were used without further purification. The reaction was carried out under an air atmosphere. Methacrylic acid (2 mmol), dipyridin-2-ylamine (1 mmol) and nickel(II)chloride (1 mmol) were added to water and the mixture was stirred for 4 h at 323 K. After cooling to room temperature, the solution was filtered. The solvent was removed from the filtrate under vacuum, and the solid residue was recrystallized from ethanol to form yellow crystals which were suitable for X-Ray diffraction study. Yield, 78%. m.p. 547 K. Analysis, calculated for C₁₈H₂₂N₃NiO_{5.50}: C 50.62, H 5.19, N 9.84; found: C 50.38, H 5.43, N 9.52. The elemental analyses were performed with a Perkin Elmer PE2400II instrument.

Refinement

Methyl H atoms were included in calculated positions with C—H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. H atoms bonded to N3 and O6 were included in calculations with N—H = 0.6 Å, O—H = 0.85 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O},\text{N})$. All other H atoms were refined independently with isotropic displacement parameters. The C—H distances refined to 0.90 (3) – 1.01 (3) Å.

Figures

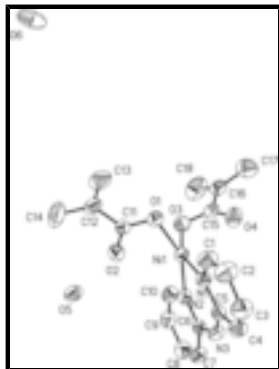


Fig. 1. The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. The H atoms are omitted.

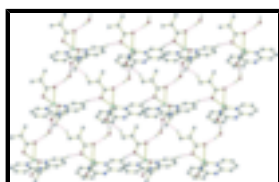


Fig. 2. The one-dimensional structure formed *via* intermolecular O—H...O and N—H...O hydrogen bonds. Hydrogen bonds are shown as red lines.

(Di-2-pyridylamine- κ^2N^1,N^1)bis(methacryato- κO)nickel(II) sesquihydrate

Crystal data

$[\text{Ni}(\text{C}_4\text{H}_5\text{O}_2)_2(\text{C}_{10}\text{H}_9\text{N}_3)] \cdot 1.5\text{H}_2\text{O}$

$M_r = 427.10$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 8.3686\ (9)\ \text{\AA}$

$b = 15.7235\ (16)\ \text{\AA}$

$c = 15.5396\ (17)\ \text{\AA}$

$\beta = 101.483\ (2)^\circ$

$V = 2003.8\ (4)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 892$

$D_x = 1.416\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 4631 reflections

$\theta = 1.9\text{--}28.3^\circ$

$\mu = 1.00\ \text{mm}^{-1}$

$T = 293\ (2)\ \text{K}$

Block, blue

$0.29 \times 0.22 \times 0.18\ \text{mm}$

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293\ (2)\ \text{K}$

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.740$, $T_{\max} = 0.872$

9960 measured reflections

3512 independent reflections

2720 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.077$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 1.9^\circ$

$h = -9 \rightarrow 9$

$k = -18 \rightarrow 17$

$l = -15 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.084$	$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$
$S = 1.00$	where $P = (F_o^2 + 2F_c^2)/3$
3512 reflections	$(\Delta/\sigma)_{\max} = 0.013$
311 parameters	$\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$
13 restraints	$\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Ni1	0.71669 (3)	0.166235 (18)	0.635377 (19)	0.05233 (13)	
O1	0.6953 (2)	0.18663 (11)	0.75859 (11)	0.0676 (5)	
O2	0.4520 (2)	0.16405 (13)	0.68304 (14)	0.0869 (6)	
O3	0.6959 (2)	0.28811 (11)	0.60684 (13)	0.0744 (5)	
O4	0.9584 (2)	0.26650 (12)	0.62783 (14)	0.0866 (6)	
O5	0.1555 (3)	0.13886 (13)	0.58041 (14)	0.0871 (6)	
O6	0.1576 (18)	0.8401 (6)	0.9099 (9)	0.365 (9)	0.50
H6A	0.2379	0.8070	0.9103	0.437*	0.50
H6B	0.0724	0.8139	0.8846	0.437*	0.50
N1	0.8388 (2)	0.05866 (11)	0.66380 (12)	0.0554 (5)	
N2	0.6341 (2)	0.13116 (12)	0.51218 (13)	0.0555 (5)	
N3	0.7837 (2)	0.00393 (12)	0.51996 (13)	0.0599 (5)	
H19	0.8113	-0.0357	0.4877	0.072*	
C1	0.9209 (4)	0.0486 (2)	0.74736 (19)	0.0786 (8)	
C2	1.0198 (4)	-0.0173 (2)	0.7755 (2)	0.0922 (10)	
C3	1.0384 (4)	-0.0799 (2)	0.7157 (2)	0.0857 (9)	

supplementary materials

C4	0.9597 (3)	-0.07131 (17)	0.63130 (19)	0.0682 (7)
C5	0.8605 (3)	-0.00130 (14)	0.60641 (16)	0.0530 (6)
C6	0.6718 (3)	0.05967 (14)	0.47463 (15)	0.0514 (5)
C7	0.5986 (3)	0.03930 (18)	0.38840 (17)	0.0659 (7)
C8	0.4874 (4)	0.0926 (2)	0.34175 (19)	0.0745 (8)
C9	0.4470 (4)	0.1665 (2)	0.38006 (19)	0.0724 (8)
C10	0.5214 (3)	0.18320 (18)	0.4632 (2)	0.0693 (7)
C11	0.5437 (3)	0.18002 (14)	0.75381 (19)	0.0612 (7)
C12	0.4763 (4)	0.18975 (16)	0.8346 (2)	0.0745 (8)
C13	0.5788 (7)	0.1974 (2)	0.9112 (3)	0.1023 (12)
C14	0.3001 (5)	0.1899 (3)	0.8252 (3)	0.1299 (14)
H13	0.2721	0.2052	0.8801	0.195*
H11	0.2583	0.1342	0.8081	0.195*
H12	0.2534	0.2304	0.7810	0.195*
C15	0.8403 (4)	0.31425 (16)	0.60955 (16)	0.0606 (6)
C16	0.8616 (4)	0.40609 (18)	0.58992 (18)	0.0745 (7)
C17	1.0179 (7)	0.4399 (3)	0.6099 (3)	0.1124 (13)
C18	0.7233 (5)	0.4528 (2)	0.5499 (3)	0.1287 (14)
H17	0.7563	0.5083	0.5343	0.193*
H18	0.6504	0.4584	0.5901	0.193*
H16	0.6688	0.4237	0.4980	0.193*
H1	0.913 (3)	0.0917 (18)	0.7842 (18)	0.083 (9)*
H2	1.086 (3)	-0.0193 (18)	0.8329 (14)	0.099 (10)*
H3	1.108 (4)	-0.131 (2)	0.733 (2)	0.117 (11)*
H4	0.964 (3)	-0.1118 (14)	0.5870 (14)	0.066 (7)*
H5	0.630 (2)	-0.0103 (11)	0.3650 (14)	0.058 (7)*
H6	0.430 (3)	0.0750 (19)	0.281 (2)	0.106 (10)*
H7	0.373 (3)	0.2059 (16)	0.3503 (17)	0.076 (8)*
H8	0.495 (3)	0.2368 (17)	0.4916 (17)	0.084 (8)*
H9	0.690 (2)	0.187 (2)	0.919 (2)	0.118 (16)*
H10	0.523 (5)	0.207 (2)	0.958 (3)	0.153 (15)*
H14	1.099 (4)	0.403 (2)	0.633 (2)	0.133 (17)*
H15	1.031 (4)	0.4937 (15)	0.592 (2)	0.134 (14)*
H20	0.257 (3)	0.1554 (17)	0.6173 (18)	0.099 (11)*
H21	0.082 (4)	0.1753 (18)	0.598 (2)	0.128 (14)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0512 (2)	0.0530 (2)	0.0534 (2)	0.00317 (13)	0.01212 (15)	-0.00762 (14)
O1	0.0629 (12)	0.0792 (12)	0.0624 (11)	-0.0013 (9)	0.0162 (9)	-0.0175 (9)
O2	0.0608 (12)	0.1171 (17)	0.0832 (14)	-0.0144 (11)	0.0152 (11)	-0.0286 (12)
O3	0.0689 (12)	0.0613 (11)	0.0941 (13)	0.0041 (9)	0.0191 (10)	-0.0042 (10)
O4	0.0766 (13)	0.0790 (13)	0.1072 (16)	0.0182 (10)	0.0254 (12)	0.0057 (11)
O5	0.0704 (13)	0.0955 (14)	0.0905 (15)	0.0182 (11)	0.0041 (12)	-0.0320 (12)
O6	0.410 (19)	0.215 (11)	0.395 (19)	-0.049 (10)	-0.096 (16)	0.069 (10)
N1	0.0556 (12)	0.0580 (12)	0.0519 (11)	0.0019 (9)	0.0089 (10)	-0.0038 (9)
N2	0.0527 (12)	0.0598 (12)	0.0546 (12)	0.0037 (10)	0.0121 (10)	0.0011 (10)

N3	0.0686 (13)	0.0582 (12)	0.0528 (12)	0.0107 (10)	0.0121 (10)	-0.0075 (10)
C1	0.084 (2)	0.086 (2)	0.0597 (18)	0.0167 (17)	-0.0003 (15)	-0.0109 (16)
C2	0.096 (2)	0.105 (2)	0.0633 (19)	0.030 (2)	-0.0122 (18)	-0.0020 (18)
C3	0.088 (2)	0.078 (2)	0.084 (2)	0.0213 (17)	-0.0027 (18)	0.0054 (18)
C4	0.0713 (17)	0.0595 (16)	0.0708 (18)	0.0055 (13)	0.0066 (15)	-0.0046 (14)
C5	0.0500 (13)	0.0518 (13)	0.0579 (15)	-0.0019 (11)	0.0125 (11)	0.0015 (12)
C6	0.0490 (13)	0.0567 (14)	0.0504 (13)	-0.0009 (11)	0.0146 (11)	0.0006 (11)
C7	0.0716 (17)	0.0738 (18)	0.0531 (16)	0.0097 (14)	0.0147 (14)	-0.0050 (14)
C8	0.0739 (19)	0.098 (2)	0.0507 (16)	-0.0005 (17)	0.0103 (15)	0.0084 (16)
C9	0.0701 (19)	0.081 (2)	0.0639 (18)	0.0116 (16)	0.0085 (15)	0.0116 (16)
C10	0.0681 (18)	0.0674 (18)	0.0710 (19)	0.0138 (14)	0.0101 (15)	0.0034 (14)
C11	0.0621 (17)	0.0529 (15)	0.0728 (18)	-0.0060 (12)	0.0234 (15)	-0.0125 (12)
C12	0.104 (2)	0.0500 (14)	0.0812 (19)	-0.0052 (14)	0.0472 (18)	-0.0095 (14)
C13	0.155 (4)	0.084 (2)	0.081 (2)	-0.020 (3)	0.056 (3)	-0.0093 (19)
C14	0.119 (3)	0.144 (3)	0.153 (4)	0.010 (2)	0.089 (3)	0.005 (3)
C15	0.0705 (18)	0.0624 (16)	0.0520 (14)	0.0061 (14)	0.0193 (14)	-0.0061 (12)
C16	0.101 (2)	0.0637 (17)	0.0669 (17)	-0.0049 (15)	0.0361 (16)	-0.0069 (14)
C17	0.147 (4)	0.093 (3)	0.104 (3)	-0.034 (3)	0.044 (3)	0.000 (2)
C18	0.160 (3)	0.080 (2)	0.161 (4)	0.039 (2)	0.067 (3)	0.030 (2)

Geometric parameters (Å, °)

Ni1—O3	1.9669 (18)	C4—H4	0.944 (16)
Ni1—N2	1.980 (2)	C6—C7	1.395 (3)
Ni1—N1	1.9801 (18)	C7—C8	1.351 (4)
Ni1—O1	1.9843 (17)	C7—H5	0.921 (15)
O1—C11	1.260 (3)	C8—C9	1.378 (4)
O2—C11	1.235 (3)	C8—H6	1.01 (3)
O3—C15	1.269 (3)	C9—C10	1.343 (4)
O4—C15	1.228 (3)	C9—H7	0.93 (3)
O5—H20	0.961 (18)	C10—H8	1.00 (3)
O5—H21	0.921 (18)	C11—C12	1.483 (4)
O6—H6A	0.8498	C12—C13	1.327 (5)
O6—H6B	0.8497	C12—C14	1.453 (4)
N1—C5	1.335 (3)	C13—H9	0.930 (18)
N1—C1	1.352 (3)	C13—H10	0.95 (4)
N2—C6	1.333 (3)	C14—H13	0.9600
N2—C10	1.361 (3)	C14—H11	0.9600
N3—C6	1.371 (3)	C14—H12	0.9600
N3—C5	1.371 (3)	C15—C16	1.494 (4)
N3—H19	0.8600	C16—C17	1.389 (5)
C1—C2	1.344 (4)	C16—C18	1.407 (4)
C1—H1	0.90 (3)	C17—H14	0.911 (18)
C2—C3	1.383 (4)	C17—H15	0.906 (19)
C2—H2	0.954 (18)	C18—H17	0.9600
C3—C4	1.353 (4)	C18—H18	0.9600
C3—H3	1.00 (3)	C18—H16	0.9600
C4—C5	1.387 (3)		
O3—Ni1—N2	93.16 (8)	C7—C8—C9	119.6 (3)

supplementary materials

O3—Ni1—N1	154.32 (8)	C7—C8—H6	118.8 (17)
N2—Ni1—N1	92.47 (8)	C9—C8—H6	121.5 (17)
O3—Ni1—O1	92.24 (8)	C10—C9—C8	118.3 (3)
N2—Ni1—O1	153.74 (8)	C10—C9—H7	118.8 (16)
N1—Ni1—O1	93.70 (7)	C8—C9—H7	122.9 (16)
C11—O1—Ni1	102.29 (16)	C9—C10—N2	123.9 (3)
C15—O3—Ni1	105.48 (16)	C9—C10—H8	119.4 (15)
H20—O5—H21	103 (3)	N2—C10—H8	116.7 (15)
H6A—O6—H6B	107.1	O2—C11—O1	120.6 (2)
C5—N1—C1	116.6 (2)	O2—C11—C12	120.0 (3)
C5—N1—Ni1	125.95 (16)	O1—C11—C12	119.3 (3)
C1—N1—Ni1	117.08 (18)	C13—C12—C14	123.5 (4)
C6—N2—C10	117.4 (2)	C13—C12—C11	118.8 (3)
C6—N2—Ni1	126.19 (16)	C14—C12—C11	117.7 (3)
C10—N2—Ni1	116.32 (18)	C12—C13—H9	124 (2)
C6—N3—C5	132.90 (19)	C12—C13—H10	112 (3)
C6—N3—H19	113.5	H9—C13—H10	124 (4)
C5—N3—H19	113.5	C12—C14—H13	109.5
C2—C1—N1	124.4 (3)	C12—C14—H11	109.5
C2—C1—H1	119.6 (17)	H13—C14—H11	109.5
N1—C1—H1	115.9 (18)	C12—C14—H12	109.5
C1—C2—C3	118.4 (3)	H13—C14—H12	109.5
C1—C2—H2	122.5 (18)	H11—C14—H12	109.5
C3—C2—H2	118.8 (17)	O4—C15—O3	121.8 (2)
C4—C3—C2	118.8 (3)	O4—C15—C16	121.0 (3)
C4—C3—H3	119 (2)	O3—C15—C16	117.2 (2)
C2—C3—H3	122 (2)	C17—C16—C18	123.6 (4)
C3—C4—C5	119.9 (3)	C17—C16—C15	118.1 (3)
C3—C4—H4	123.9 (15)	C18—C16—C15	118.3 (3)
C5—C4—H4	116.1 (15)	C16—C17—H14	116 (3)
N1—C5—N3	120.8 (2)	C16—C17—H15	117 (3)
N1—C5—C4	121.9 (2)	H14—C17—H15	126 (4)
N3—C5—C4	117.3 (2)	C16—C18—H17	109.5
N2—C6—N3	120.8 (2)	C16—C18—H18	109.5
N2—C6—C7	121.1 (2)	H17—C18—H18	109.5
N3—C6—C7	118.1 (2)	C16—C18—H16	109.5
C8—C7—C6	119.8 (3)	H17—C18—H16	109.5
C8—C7—H5	122.3 (14)	H18—C18—H16	109.5
C6—C7—H5	117.9 (14)		
O3—Ni1—O1—C11	90.93 (15)	C6—N3—C5—C4	175.2 (2)
N2—Ni1—O1—C11	-10.8 (3)	C3—C4—C5—N1	-0.4 (4)
N1—Ni1—O1—C11	-114.07 (15)	C3—C4—C5—N3	-179.9 (3)
N2—Ni1—O3—C15	-105.33 (17)	C10—N2—C6—N3	-179.7 (2)
N1—Ni1—O3—C15	-2.9 (3)	Ni1—N2—C6—N3	-3.6 (3)
O1—Ni1—O3—C15	100.38 (17)	C10—N2—C6—C7	0.1 (3)
O3—Ni1—N1—C5	-94.3 (2)	Ni1—N2—C6—C7	176.25 (17)
N2—Ni1—N1—C5	8.28 (19)	C5—N3—C6—N2	9.6 (4)
O1—Ni1—N1—C5	162.74 (18)	C5—N3—C6—C7	-170.3 (2)
O3—Ni1—N1—C1	78.9 (3)	N2—C6—C7—C8	0.0 (4)

N2—Ni1—N1—C1	-178.6 (2)	N3—C6—C7—C8	179.8 (2)
O1—Ni1—N1—C1	-24.1 (2)	C6—C7—C8—C9	-0.3 (4)
O3—Ni1—N2—C6	151.52 (18)	C7—C8—C9—C10	0.5 (4)
N1—Ni1—N2—C6	-3.41 (19)	C8—C9—C10—N2	-0.4 (4)
O1—Ni1—N2—C6	-106.9 (2)	C6—N2—C10—C9	0.1 (4)
O3—Ni1—N2—C10	-32.28 (19)	Ni1—N2—C10—C9	-176.4 (2)
N1—Ni1—N2—C10	172.79 (18)	Ni1—O1—C11—O2	0.2 (3)
O1—Ni1—N2—C10	69.3 (3)	Ni1—O1—C11—C12	178.91 (18)
C5—N1—C1—C2	-0.7 (4)	O2—C11—C12—C13	172.6 (3)
Ni1—N1—C1—C2	-174.4 (3)	O1—C11—C12—C13	-6.1 (4)
N1—C1—C2—C3	-0.7 (5)	O2—C11—C12—C14	-7.1 (4)
C1—C2—C3—C4	1.6 (5)	O1—C11—C12—C14	174.2 (3)
C2—C3—C4—C5	-1.1 (5)	Ni1—O3—C15—O4	-0.5 (3)
C1—N1—C5—N3	-179.3 (2)	Ni1—O3—C15—C16	179.79 (18)
Ni1—N1—C5—N3	-6.1 (3)	O4—C15—C16—C17	-12.7 (4)
C1—N1—C5—C4	1.2 (4)	O3—C15—C16—C17	167.0 (3)
Ni1—N1—C5—C4	174.35 (18)	O4—C15—C16—C18	165.4 (3)
C6—N3—C5—N1	-4.3 (4)	O3—C15—C16—C18	-14.9 (4)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O6—H6B \cdots O2 ⁱ	0.85	2.57	3.172	129
O6—H6B \cdots O3 ⁱ	0.85	2.31	3.030	143
N3—H19 \cdots O5 ⁱⁱ	0.86	1.99	2.837 (3)	170
O5—H20 \cdots O2	0.96 (3)	1.75 (3)	2.698 (3)	169 (2)
O5—H21 \cdots O4 ⁱⁱⁱ	0.92 (3)	1.88 (3)	2.789 (3)	169 (3)

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

Fig. 1

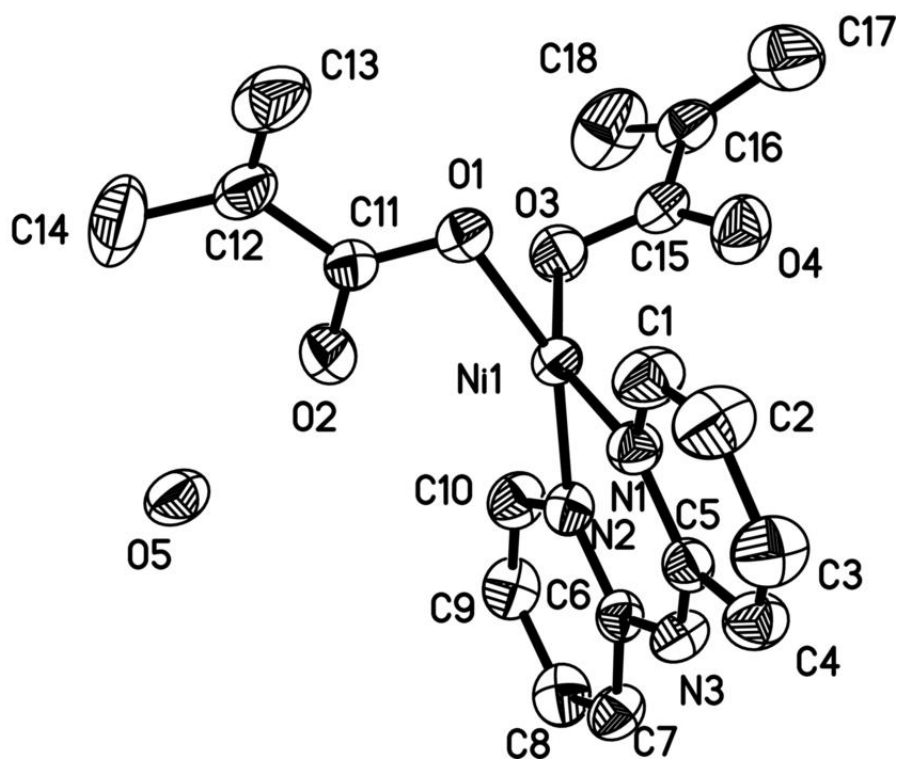


Fig. 2

