Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Bis[(3-hydroxy-2-naphthyl)(2-pyridylmethylenehydrazono)methanolato]-nickel(II)

**Wenjun Kang,[a] Shizhu Sun,[b] Dacheng Li,[a] Daqi Wang[a] and Jianmin Dou[a]***

[a]School of Chemistry and Chemical Engineering, Liaocheng University, Liaocheng 252059, People's Republic of China, and [b]Department of Adult Education, Dongying Vocation College, Dongying 257091, People's Republic of China
Correspondence e-mail: jmdou@lcu.edu.cn

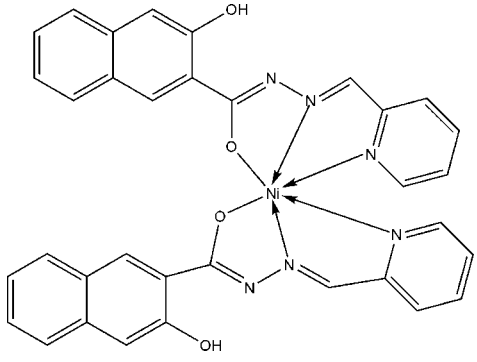
Received 26 September 2007; accepted 28 September 2007

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.047; $wR$ factor = 0.148; data-to-parameter ratio = 12.8.

In the title complex, $[Ni(C_{17}H_{12}N_3O_2)_2]$, the $Ni^{II}$ ion is coordinated by two O [Ni—O = 2.066 (3) and 2.068 (3) Å] and four N [Ni—N = 1.982 (3)–2.120 (3)Å] atoms in a distorted octahedral geometry. Two intramolecular O—H···N hydrogen bonds contribute to the molecular conformation. Weak intermolecular C—H···O hydrogen bonds stabilize the crystal packing.

## Related literature

For related crystal structures of Cu and Ni complexes with aroylhydrazone derivatives, see Liu *et al.* (2005*a*,*b*). For general background, see Beghidja *et al.* (2006), and for the specific biological activities of aroylhydrazones, see Mostafa & Haifaa (2007).




## Experimental

### Crystal data

$[Ni(C_{17}H_{12}N_3O_2)_2]$
$M_r$ = 639.30
Monoclinic, $P2_1/c$
$a$ = 21.257 (3) Å
$b$ = 11.653 (3) Å
$c$ = 11.938 (3) Å
$\beta$ = 91.686 (3)°
$V$ = 2955.6 (10) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.71 mm$^{-1}$
$T$ = 293 (2) K
0.69 × 0.66 × 0.13 mm

### Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.641, $T_{max}$ = 0.914
14859 measured reflections
5202 independent reflections
3068 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.089

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.149
$S$ = 1.01
5202 reflections
408 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.45 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O4—H4···N4 | 0.82 | 1.83 | 2.561 (4) | 148 |
| O2—H2···N1 | 0.82 | 1.84 | 2.567 (4) | 147 |
| C15—H15···O4$^{i}$ | 0.93 | 2.56 | 3.195 (6) | 126 |
| C32—H32···O2$^{ii}$ | 0.93 | 2.38 | 3.295 (6) | 168 |

Symmetry codes: (i) $x, y+1, z$; (ii) $x, y, z+1$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge the support of the National Natural Science Foundation of China, grant No. 20671048.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2309).

## References

Beghidja, C., Rogez, G., Kortus, J., Wesolek, M. & Welter, R. (2006). *J. Am. Chem. Soc.* **128**, 3140–3141.
Liu, M.-L., Dou, J.-M., Wang, D.-Q. & Li, D.-C. (2005*a*). *Acta Cryst.* E**61**, m1662–m1664.
Liu, M.-L., Dou, J.-M., Wang, D.-Q. & Li, D.-C. (2005*b*). *Acta Cryst.* E**61**, m1366–m1367.
Mostafa, E. B. & Haifaa, E. T. (2007). *Spectrochim. Acta Part A*, **66**, 28–36.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

 doi:10.1107/S1600536807047770  

# supplementary materials

*Acta Cryst.* (2007). E**63**, m2667 [ **doi:10.1107/S1600536807047770** ]

# Bis[(3-hydroxy-2-naphthyl)(2-pyridylmethylenehydrazono)methanolato]nickel(II)

**W. Kang, S. Sun, D. Li, D. Wang and J. Dou**

## Comment

The chemistry of aroylhydrazones is widely studied due to their coordination abilities to metal ions (Beghidja *et al.*, 2006) and their biological activities (Mostafa & Haifaa, 2007). Our group have reported the crystal structures of two aroylhydrazone complexes - $[Cu(C_{12}H_{12}N_2O_3)(C_5H_5N)]$ (Liu *et al.*, 2005*a*) and $[Ni(C_{18}H_{12}N_2O_3)(C_5H_5N)]$ (Liu *et al.*, 2005*b*). As an extension of our work on the structural characterization of aroylhydrazone derivatives, the title complex, (I), was synthesized and characterized.

In (I) (Fig. 1), the Ni ion is six-coordinated by four N atoms and two O atoms in a distorted octahedron geometry. Each ligand coordinate the Ni ion in a tridentate mode *via* N atom of the azomethine group, O atom of the the hydroxyl group and N atom of the pyridine ring. Each ligand forms two five-membered chelate rings Ni—N—N—C—O and Ni—N—C—C—N with the nickel atom. The atoms N3, N6, O3 and O1 constitute the equatorial plane, with the mean deviation from plane of 0.43 (6) Å. The angles O1—Ni1—N3 and O3—Ni1—N6 are 155.95 (12) and 155.93 (13)°, respectively. The axial positions are occupied by the atoms N5 and N2. The angle of N5—Ni1—N2 is 176.33 (14)°. There are two intramolecular O2—H2···N1 and O4—H4···N4 hydrogen bonds (Table 1 and Fig. 1).

In the crystal, the weak intermolecular C—H···O hydrogen bonds (Table 1) contribute to the crystal packing stabilization.

## Experimental

A dimethylformamide solution (10 ml) of $H_2L$ (0.073 g, 0.25 mmol) and KOH (0.014 g, 0.25 mmol) was mixed with a pyridine solution (10 ml) of $Ni(OAC)_2{}^4H_2O$ (0.062 g, 0.25 mmol). The mixture was stirred for 4 h, filtered, and was left to stand. Two weeks later, red crystals were isolated from the solution (m.p. >573 K). Analysis, calculated for $C_{34}H_{24}N_6NiO_4$: C 63.88, H 3.78, N 13.15%; found: C 63.76, H 3.80, N 13.01%.

## Refinement

All H atoms were placed in geometrically idealized positions (O—H 0.82 and C—H 0.93 Å) and treated as riding on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ and $U_{iso}(H) = 1.5U_{eq}(O)$.

## Figures

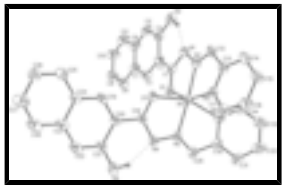


Fig. 1. The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. The dashed lines denote hydrogen bonds. C-bound H atoms have been omitted for clarity.

**Bis[(3-hydroxy-2-naphthyl)(2-pyridylmethylenehydrazono)methanolato]nickel(II)**

*Crystal data*

| | |
|---|---|
| $[Ni(C_{17}H_{12}N_3O_2)_2]$ | $F_{000}$ = 1320 |
| $M_r$ = 639.30 | $D_x$ = 1.437 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| $a$ = 21.257 (3) Å | Cell parameters from 3186 reflections |
| $b$ = 11.653 (3) Å | θ = 2.4–22.9º |
| $c$ = 11.938 (3) Å | μ = 0.71 mm$^{-1}$ |
| β = 91.686 (3)º | $T$ = 293 (2) K |
| $V$ = 2955.6 (10) Å$^3$ | Plate, red |
| $Z$ = 4 | 0.69 × 0.66 × 0.13 mm |

*Data collection*

| | |
|---|---|
| Siemens SMART CCD area-detector diffractometer | 5202 independent reflections |
| Radiation source: fine-focus sealed tube | 3068 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.089 |
| $T$ = 293(2) K | $\theta_{max}$ = 25.0º |
| φ and ω scans | $\theta_{min}$ = 1.9º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h$ = −25→23 |
| $T_{min}$ = 0.641, $T_{max}$ = 0.914 | $k$ = −13→9 |
| 14859 measured reflections | $l$ = −14→13 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.047 | H-atom parameters constrained |
| $wR(F^2)$ = 0.149 | $w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 0.6517P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.01 | $(\Delta/\sigma)_{max}$ = 0.001 |
| 5202 reflections | $\Delta\rho_{max}$ = 0.34 e Å$^{-3}$ |
| 408 parameters | $\Delta\rho_{min}$ = −0.45 e Å$^{-3}$ |
| 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 $F^2 > 2\mathrm{sigma}(F^2)$ 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 | 0.24926 (2) | 0.45673 (5) | 1.00715 (4) | 0.03962 (19) |
| N1 | 0.31082 (16) | 0.5566 (3) | 0.8193 (3) | 0.0468 (9) |
| N2 | 0.26876 (15) | 0.5813 (3) | 0.9004 (3) | 0.0433 (8) |
| N3 | 0.18729 (15) | 0.5883 (3) | 1.0576 (3) | 0.0405 (8) |
| N4 | 0.19264 (15) | 0.2411 (3) | 1.0620 (3) | 0.0444 (9) |
| N5 | 0.23030 (15) | 0.3252 (3) | 1.1058 (3) | 0.0428 (8) |
| N6 | 0.30319 (16) | 0.4982 (3) | 1.1532 (3) | 0.0480 (9) |
| O1 | 0.31986 (13) | 0.3844 (3) | 0.9151 (2) | 0.0511 (8) |
| O2 | 0.37269 (18) | 0.5822 (3) | 0.6396 (3) | 0.0745 (10) |
| H2 | 0.3465 | 0.5973 | 0.6865 | 0.112* |
| O3 | 0.18427 (12) | 0.3650 (2) | 0.9110 (2) | 0.0435 (7) |
| O4 | 0.12798 (17) | 0.0556 (2) | 1.0564 (3) | 0.0663 (9) |
| H4 | 0.1537 | 0.1029 | 1.0800 | 0.099* |
| C1 | 0.33299 (18) | 0.4505 (4) | 0.8363 (3) | 0.0413 (10) |
| C2 | 0.4035 (2) | 0.3014 (4) | 0.7651 (3) | 0.0512 (12) |
| H2A | 0.3924 | 0.2582 | 0.8271 | 0.061* |
| C3 | 0.37842 (18) | 0.4088 (4) | 0.7523 (3) | 0.0436 (10) |
| C4 | 0.3953 (2) | 0.4740 (4) | 0.6578 (3) | 0.0541 (12) |
| C5 | 0.4366 (2) | 0.4292 (5) | 0.5824 (4) | 0.0639 (14) |
| H5 | 0.4480 | 0.4735 | 0.5215 | 0.077* |
| C6 | 0.4619 (2) | 0.3181 (5) | 0.5953 (4) | 0.0575 (13) |
| C7 | 0.44529 (19) | 0.2531 (4) | 0.6885 (4) | 0.0533 (12) |
| C8 | 0.4700 (2) | 0.1419 (5) | 0.7015 (4) | 0.0710 (15) |
| H8 | 0.4593 | 0.0984 | 0.7634 | 0.085* |
| C9 | 0.5087 (3) | 0.0976 (6) | 0.6261 (5) | 0.0848 (18) |
| H9 | 0.5241 | 0.0235 | 0.6353 | 0.102* |
| C10 | 0.5258 (2) | 0.1631 (6) | 0.5335 (5) | 0.0867 (19) |
| H10 | 0.5533 | 0.1324 | 0.4821 | 0.104* |
| C11 | 0.5032 (2) | 0.2693 (6) | 0.5180 (4) | 0.0785 (17) |
| H11 | 0.5148 | 0.3114 | 0.4557 | 0.094* |
| C12 | 0.2399 (2) | 0.6765 (4) | 0.9079 (4) | 0.0521 (12) |
| H12 | 0.2462 | 0.7362 | 0.8577 | 0.063* |
| C13 | 0.19617 (19) | 0.6866 (4) | 1.0007 (3) | 0.0469 (11) |
| C14 | 0.1689 (2) | 0.7886 (4) | 1.0314 (4) | 0.0656 (14) |
| H14 | 0.1760 | 0.8553 | 0.9909 | 0.079* |
| C15 | 0.1311 (2) | 0.7901 (4) | 1.1229 (4) | 0.0689 (14) |
| H15 | 0.1126 | 0.8581 | 1.1457 | 0.083* |
| C16 | 0.1211 (2) | 0.6909 (4) | 1.1798 (4) | 0.0604 (13) |

| | | | | |
|---|---|---|---|---|
| H16 | 0.0954 | 0.6903 | 1.2416 | 0.072* |
| C17 | 0.14953 (19) | 0.5914 (4) | 1.1450 (3) | 0.0508 (11) |
| H17 | 0.1421 | 0.5238 | 1.1839 | 0.061* |
| C18 | 0.17036 (18) | 0.2725 (4) | 0.9597 (3) | 0.0388 (10) |
| C19 | 0.10074 (18) | 0.2221 (3) | 0.8001 (3) | 0.0417 (10) |
| H19 | 0.1155 | 0.2878 | 0.7654 | 0.050* |
| C20 | 0.12551 (18) | 0.1931 (3) | 0.9040 (3) | 0.0381 (10) |
| C21 | 0.1047 (2) | 0.0897 (4) | 0.9549 (4) | 0.0485 (11) |
| C22 | 0.0590 (2) | 0.0251 (4) | 0.9018 (4) | 0.0555 (12) |
| H22 | 0.0456 | −0.0422 | 0.9354 | 0.067* |
| C23 | 0.0318 (2) | 0.0575 (4) | 0.7981 (4) | 0.0479 (11) |
| C24 | 0.05415 (19) | 0.1566 (3) | 0.7443 (3) | 0.0434 (10) |
| C25 | 0.0279 (2) | 0.1874 (4) | 0.6383 (4) | 0.0554 (12) |
| H25 | 0.0428 | 0.2519 | 0.6017 | 0.066* |
| C26 | −0.0189 (2) | 0.1237 (5) | 0.5895 (4) | 0.0650 (14) |
| H26 | −0.0357 | 0.1442 | 0.5195 | 0.078* |
| C27 | −0.0418 (2) | 0.0276 (5) | 0.6446 (5) | 0.0732 (15) |
| H27 | −0.0746 | −0.0142 | 0.6116 | 0.088* |
| C28 | −0.0173 (2) | −0.0059 (4) | 0.7443 (5) | 0.0649 (14) |
| H28 | −0.0327 | −0.0715 | 0.7784 | 0.078* |
| C29 | 0.25446 (19) | 0.3233 (4) | 1.2053 (3) | 0.0503 (12) |
| H29 | 0.2491 | 0.2617 | 1.2536 | 0.060* |
| C30 | 0.2909 (2) | 0.4242 (4) | 1.2374 (3) | 0.0514 (12) |
| C31 | 0.3103 (2) | 0.4486 (5) | 1.3469 (4) | 0.0744 (16) |
| H31 | 0.3017 | 0.3979 | 1.4046 | 0.089* |
| C32 | 0.3428 (3) | 0.5496 (6) | 1.3691 (5) | 0.090 (2) |
| H32 | 0.3554 | 0.5680 | 1.4422 | 0.108* |
| C33 | 0.3562 (3) | 0.6220 (5) | 1.2828 (5) | 0.0805 (17) |
| H33 | 0.3788 | 0.6893 | 1.2959 | 0.097* |
| C34 | 0.3356 (2) | 0.5933 (5) | 1.1769 (4) | 0.0618 (13) |
| H34 | 0.3448 | 0.6427 | 1.1184 | 0.074* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ni1 | 0.0449 (3) | 0.0372 (3) | 0.0370 (3) | 0.0009 (3) | 0.0062 (2) | 0.0018 (2) |
| N1 | 0.052 (2) | 0.046 (2) | 0.043 (2) | −0.0032 (19) | 0.0177 (17) | 0.0012 (17) |
| N2 | 0.047 (2) | 0.042 (2) | 0.0417 (19) | −0.0034 (19) | 0.0059 (16) | −0.0001 (17) |
| N3 | 0.0388 (18) | 0.039 (2) | 0.0438 (19) | 0.0003 (18) | 0.0075 (15) | 0.0060 (17) |
| N4 | 0.049 (2) | 0.038 (2) | 0.047 (2) | 0.0005 (19) | 0.0070 (16) | 0.0088 (17) |
| N5 | 0.045 (2) | 0.045 (2) | 0.039 (2) | 0.0068 (19) | 0.0072 (16) | 0.0055 (17) |
| N6 | 0.046 (2) | 0.056 (2) | 0.042 (2) | 0.004 (2) | 0.0039 (16) | −0.0093 (19) |
| O1 | 0.0570 (18) | 0.0515 (19) | 0.0453 (16) | 0.0092 (16) | 0.0111 (14) | 0.0083 (15) |
| O2 | 0.101 (3) | 0.071 (2) | 0.054 (2) | 0.018 (2) | 0.0344 (18) | 0.0169 (18) |
| O3 | 0.0544 (17) | 0.0339 (16) | 0.0422 (16) | −0.0044 (15) | 0.0016 (13) | 0.0072 (13) |
| O4 | 0.102 (3) | 0.0379 (19) | 0.059 (2) | −0.0121 (19) | 0.0050 (18) | 0.0136 (16) |
| C1 | 0.041 (2) | 0.045 (3) | 0.039 (2) | −0.001 (2) | 0.0021 (18) | −0.002 (2) |
| C2 | 0.051 (3) | 0.055 (3) | 0.047 (3) | −0.001 (3) | 0.005 (2) | −0.003 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C3 | 0.041 (2) | 0.054 (3) | 0.036 (2) | −0.002 (2) | 0.0038 (18) | −0.004 (2) |
| C4 | 0.061 (3) | 0.061 (3) | 0.041 (2) | 0.007 (3) | 0.011 (2) | 0.003 (2) |
| C5 | 0.071 (3) | 0.081 (4) | 0.041 (3) | 0.002 (3) | 0.018 (2) | 0.002 (3) |
| C6 | 0.046 (3) | 0.081 (4) | 0.046 (3) | 0.006 (3) | 0.006 (2) | −0.011 (3) |
| C7 | 0.041 (3) | 0.068 (3) | 0.051 (3) | 0.007 (3) | 0.002 (2) | −0.010 (3) |
| C8 | 0.072 (3) | 0.074 (4) | 0.068 (3) | 0.023 (3) | 0.003 (3) | −0.009 (3) |
| C9 | 0.074 (4) | 0.090 (5) | 0.090 (4) | 0.031 (4) | −0.001 (3) | −0.022 (4) |
| C10 | 0.063 (3) | 0.119 (6) | 0.079 (4) | 0.028 (4) | 0.010 (3) | −0.035 (4) |
| C11 | 0.066 (3) | 0.111 (5) | 0.060 (3) | 0.015 (4) | 0.019 (3) | −0.014 (3) |
| C12 | 0.061 (3) | 0.039 (3) | 0.058 (3) | 0.002 (3) | 0.019 (2) | 0.011 (2) |
| C13 | 0.047 (3) | 0.042 (3) | 0.052 (3) | 0.006 (2) | 0.009 (2) | 0.002 (2) |
| C14 | 0.082 (4) | 0.037 (3) | 0.079 (3) | 0.008 (3) | 0.028 (3) | 0.008 (3) |
| C15 | 0.079 (3) | 0.048 (3) | 0.081 (4) | 0.018 (3) | 0.028 (3) | −0.005 (3) |
| C16 | 0.063 (3) | 0.060 (3) | 0.060 (3) | 0.009 (3) | 0.022 (2) | −0.002 (3) |
| C17 | 0.054 (3) | 0.044 (3) | 0.055 (3) | −0.004 (2) | 0.015 (2) | 0.002 (2) |
| C18 | 0.044 (2) | 0.033 (2) | 0.040 (2) | 0.009 (2) | 0.0140 (19) | 0.004 (2) |
| C19 | 0.044 (2) | 0.030 (2) | 0.052 (3) | −0.002 (2) | 0.015 (2) | 0.002 (2) |
| C20 | 0.043 (2) | 0.026 (2) | 0.046 (2) | 0.000 (2) | 0.0141 (19) | −0.0025 (19) |
| C21 | 0.065 (3) | 0.028 (2) | 0.052 (3) | 0.000 (2) | 0.019 (2) | 0.004 (2) |
| C22 | 0.067 (3) | 0.029 (3) | 0.072 (3) | −0.015 (2) | 0.025 (3) | −0.001 (2) |
| C23 | 0.049 (3) | 0.034 (3) | 0.061 (3) | −0.003 (2) | 0.020 (2) | −0.012 (2) |
| C24 | 0.043 (2) | 0.033 (2) | 0.055 (3) | 0.000 (2) | 0.012 (2) | −0.010 (2) |
| C25 | 0.054 (3) | 0.048 (3) | 0.065 (3) | −0.006 (3) | 0.004 (2) | −0.005 (2) |
| C26 | 0.063 (3) | 0.064 (4) | 0.067 (3) | 0.002 (3) | −0.007 (3) | −0.012 (3) |
| C27 | 0.060 (3) | 0.068 (4) | 0.092 (4) | −0.015 (3) | 0.007 (3) | −0.026 (3) |
| C28 | 0.065 (3) | 0.046 (3) | 0.085 (4) | −0.016 (3) | 0.020 (3) | −0.012 (3) |
| C29 | 0.054 (3) | 0.054 (3) | 0.043 (3) | 0.010 (3) | 0.008 (2) | 0.012 (2) |
| C30 | 0.050 (3) | 0.065 (3) | 0.039 (2) | 0.014 (3) | 0.003 (2) | 0.003 (2) |
| C31 | 0.085 (4) | 0.095 (5) | 0.042 (3) | 0.018 (4) | −0.010 (2) | 0.000 (3) |
| C32 | 0.096 (4) | 0.108 (5) | 0.064 (4) | 0.028 (4) | −0.033 (3) | −0.029 (4) |
| C33 | 0.085 (4) | 0.077 (4) | 0.078 (4) | 0.009 (3) | −0.022 (3) | −0.026 (3) |
| C34 | 0.058 (3) | 0.063 (3) | 0.064 (3) | −0.001 (3) | −0.003 (2) | −0.012 (3) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Ni1—N5 | 1.982 (3) | C11—H11 | 0.9300 |
| Ni1—N2 | 1.983 (3) | C12—C13 | 1.472 (5) |
| Ni1—O1 | 2.066 (3) | C12—H12 | 0.9300 |
| Ni1—O3 | 2.068 (3) | C13—C14 | 1.376 (6) |
| Ni1—N6 | 2.114 (3) | C14—C15 | 1.376 (6) |
| Ni1—N3 | 2.120 (3) | C14—H14 | 0.9300 |
| N1—C1 | 1.336 (5) | C15—C16 | 1.360 (6) |
| N1—N2 | 1.368 (4) | C15—H15 | 0.9300 |
| N2—C12 | 1.272 (5) | C16—C17 | 1.377 (6) |
| N3—C17 | 1.336 (5) | C16—H16 | 0.9300 |
| N3—C13 | 1.347 (5) | C17—H17 | 0.9300 |
| N4—C18 | 1.349 (5) | C18—C20 | 1.473 (5) |
| N4—N5 | 1.360 (4) | C19—C20 | 1.375 (5) |
| N5—C29 | 1.280 (5) | C19—C24 | 1.403 (5) |

| | | | |
|---|---|---|---|
| N6—C34 | 1.331 (6) | C19—H19 | 0.9300 |
| N6—C30 | 1.355 (5) | C20—C21 | 1.426 (5) |
| O1—C1 | 1.254 (5) | C21—C22 | 1.370 (6) |
| O2—C4 | 1.364 (5) | C22—C23 | 1.401 (6) |
| O2—H2 | 0.8200 | C22—H22 | 0.9300 |
| O3—C18 | 1.263 (4) | C23—C24 | 1.410 (6) |
| O4—C21 | 1.354 (5) | C23—C28 | 1.417 (6) |
| O4—H4 | 0.8200 | C24—C25 | 1.413 (6) |
| C1—C3 | 1.494 (5) | C25—C26 | 1.360 (6) |
| C2—C3 | 1.367 (6) | C25—H25 | 0.9300 |
| C2—C7 | 1.411 (6) | C26—C27 | 1.392 (7) |
| C2—H2A | 0.9300 | C26—H26 | 0.9300 |
| C3—C4 | 1.415 (6) | C27—C28 | 1.343 (7) |
| C4—C5 | 1.378 (6) | C27—H27 | 0.9300 |
| C5—C6 | 1.409 (6) | C28—H28 | 0.9300 |
| C5—H5 | 0.9300 | C29—C30 | 1.454 (6) |
| C6—C7 | 1.399 (6) | C29—H29 | 0.9300 |
| C6—C11 | 1.412 (6) | C30—C31 | 1.389 (6) |
| C7—C8 | 1.405 (6) | C31—C32 | 1.385 (7) |
| C8—C9 | 1.340 (7) | C31—H31 | 0.9300 |
| C8—H8 | 0.9300 | C32—C33 | 1.368 (8) |
| C9—C10 | 1.401 (8) | C32—H32 | 0.9300 |
| C9—H9 | 0.9300 | C33—C34 | 1.367 (6) |
| C10—C11 | 1.339 (7) | C33—H33 | 0.9300 |
| C10—H10 | 0.9300 | C34—H34 | 0.9300 |
| N5—Ni1—N2 | 176.33 (14) | C13—C12—H12 | 122.0 |
| N5—Ni1—O1 | 99.45 (12) | N3—C13—C14 | 122.3 (4) |
| N2—Ni1—O1 | 77.76 (13) | N3—C13—C12 | 114.3 (4) |
| N5—Ni1—O3 | 77.62 (13) | C14—C13—C12 | 123.3 (4) |
| N2—Ni1—O3 | 99.83 (12) | C13—C14—C15 | 118.8 (4) |
| O1—Ni1—O3 | 88.73 (11) | C13—C14—H14 | 120.6 |
| N5—Ni1—N6 | 78.55 (15) | C15—C14—H14 | 120.6 |
| N2—Ni1—N6 | 104.12 (14) | C16—C15—C14 | 119.3 (5) |
| O1—Ni1—N6 | 98.46 (12) | C16—C15—H15 | 120.4 |
| O3—Ni1—N6 | 155.94 (13) | C14—C15—H15 | 120.4 |
| N5—Ni1—N3 | 104.59 (13) | C15—C16—C17 | 119.2 (4) |
| N2—Ni1—N3 | 78.25 (13) | C15—C16—H16 | 120.4 |
| O1—Ni1—N3 | 155.95 (12) | C17—C16—H16 | 120.4 |
| O3—Ni1—N3 | 96.94 (12) | N3—C17—C16 | 122.5 (4) |
| N6—Ni1—N3 | 85.81 (13) | N3—C17—H17 | 118.7 |
| C1—N1—N2 | 108.8 (3) | C16—C17—H17 | 118.7 |
| C12—N2—N1 | 124.0 (4) | O3—C18—N4 | 124.6 (4) |
| C12—N2—Ni1 | 118.9 (3) | O3—C18—C20 | 119.1 (4) |
| N1—N2—Ni1 | 117.0 (3) | N4—C18—C20 | 116.3 (4) |
| C17—N3—C13 | 117.8 (4) | C20—C19—C24 | 122.8 (4) |
| C17—N3—Ni1 | 129.3 (3) | C20—C19—H19 | 118.6 |
| C13—N3—Ni1 | 112.0 (2) | C24—C19—H19 | 118.6 |
| C18—N4—N5 | 109.8 (3) | C19—C20—C21 | 118.4 (4) |
| C29—N5—N4 | 124.0 (4) | C19—C20—C18 | 118.6 (4) |

| | | | |
|---|---|---|---|
| C29—N5—Ni1 | 118.8 (3) | C21—C20—C18 | 122.9 (4) |
| N4—N5—Ni1 | 117.2 (2) | O4—C21—C22 | 119.3 (4) |
| C34—N6—C30 | 118.8 (4) | O4—C21—C20 | 121.3 (4) |
| C34—N6—Ni1 | 129.1 (3) | C22—C21—C20 | 119.4 (4) |
| C30—N6—Ni1 | 110.8 (3) | C21—C22—C23 | 121.9 (4) |
| C1—O1—Ni1 | 109.2 (3) | C21—C22—H22 | 119.0 |
| C4—O2—H2 | 109.5 | C23—C22—H22 | 119.0 |
| C18—O3—Ni1 | 110.3 (2) | C22—C23—C24 | 119.2 (4) |
| C21—O4—H4 | 109.5 | C22—C23—C28 | 122.6 (4) |
| O1—C1—N1 | 126.7 (4) | C24—C23—C28 | 118.2 (4) |
| O1—C1—C3 | 117.8 (4) | C19—C24—C23 | 118.1 (4) |
| N1—C1—C3 | 115.6 (4) | C19—C24—C25 | 122.8 (4) |
| C3—C2—C7 | 122.8 (4) | C23—C24—C25 | 119.1 (4) |
| C3—C2—H2A | 118.6 | C26—C25—C24 | 120.6 (5) |
| C7—C2—H2A | 118.6 | C26—C25—H25 | 119.7 |
| C2—C3—C4 | 118.3 (4) | C24—C25—H25 | 119.7 |
| C2—C3—C1 | 118.7 (4) | C25—C26—C27 | 119.9 (5) |
| C4—C3—C1 | 123.0 (4) | C25—C26—H26 | 120.1 |
| O2—C4—C5 | 118.3 (4) | C27—C26—H26 | 120.1 |
| O2—C4—C3 | 121.8 (4) | C28—C27—C26 | 121.4 (5) |
| C5—C4—C3 | 120.0 (5) | C28—C27—H27 | 119.3 |
| C4—C5—C6 | 121.7 (4) | C26—C27—H27 | 119.3 |
| C4—C5—H5 | 119.2 | C27—C28—C23 | 120.8 (5) |
| C6—C5—H5 | 119.2 | C27—C28—H28 | 119.6 |
| C7—C6—C5 | 118.7 (4) | C23—C28—H28 | 119.6 |
| C7—C6—C11 | 118.6 (5) | N5—C29—C30 | 115.3 (4) |
| C5—C6—C11 | 122.8 (5) | N5—C29—H29 | 122.3 |
| C6—C7—C8 | 119.0 (4) | C30—C29—H29 | 122.3 |
| C6—C7—C2 | 118.6 (4) | N6—C30—C31 | 120.7 (5) |
| C8—C7—C2 | 122.4 (5) | N6—C30—C29 | 115.6 (4) |
| C9—C8—C7 | 121.0 (5) | C31—C30—C29 | 123.7 (5) |
| C9—C8—H8 | 119.5 | C32—C31—C30 | 119.1 (5) |
| C7—C8—H8 | 119.5 | C32—C31—H31 | 120.5 |
| C8—C9—C10 | 120.0 (6) | C30—C31—H31 | 120.5 |
| C8—C9—H9 | 120.0 | C33—C32—C31 | 119.6 (5) |
| C10—C9—H9 | 120.0 | C33—C32—H32 | 120.2 |
| C11—C10—C9 | 120.7 (5) | C31—C32—H32 | 120.2 |
| C11—C10—H10 | 119.6 | C34—C33—C32 | 118.5 (6) |
| C9—C10—H10 | 119.6 | C34—C33—H33 | 120.8 |
| C10—C11—C6 | 120.7 (5) | C32—C33—H33 | 120.8 |
| C10—C11—H11 | 119.7 | N6—C34—C33 | 123.4 (5) |
| C6—C11—H11 | 119.7 | N6—C34—H34 | 118.3 |
| N2—C12—C13 | 116.1 (4) | C33—C34—H34 | 118.3 |
| N2—C12—H12 | 122.0 | | |

| | | | |
|---|---|---|---|
| C1—N1—N2—C12 | 179.2 (4) | C11—C6—C7—C8 | −0.3 (7) |
| C1—N1—N2—Ni1 | −4.0 (4) | C5—C6—C7—C2 | 0.6 (6) |
| N5—Ni1—N2—C12 | 142 (2) | C11—C6—C7—C2 | −179.3 (4) |
| O1—Ni1—N2—C12 | −177.3 (4) | C3—C2—C7—C6 | 0.0 (6) |
| O3—Ni1—N2—C12 | 96.1 (3) | C3—C2—C7—C8 | −179.0 (4) |

| | | | |
|---|---|---|---|
| N6—Ni1—N2—C12 | −81.6 (3) | C6—C7—C8—C9 | −0.3 (7) |
| N3—Ni1—N2—C12 | 1.0 (3) | C2—C7—C8—C9 | 178.7 (5) |
| N5—Ni1—N2—N1 | −35 (2) | C7—C8—C9—C10 | 1.0 (8) |
| O1—Ni1—N2—N1 | 5.7 (3) | C8—C9—C10—C11 | −1.1 (9) |
| O3—Ni1—N2—N1 | −80.9 (3) | C9—C10—C11—C6 | 0.5 (8) |
| N6—Ni1—N2—N1 | 101.4 (3) | C7—C6—C11—C10 | 0.2 (7) |
| N3—Ni1—N2—N1 | −176.0 (3) | C5—C6—C11—C10 | −179.7 (5) |
| N5—Ni1—N3—C17 | 8.4 (4) | N1—N2—C12—C13 | 179.6 (3) |
| N2—Ni1—N3—C17 | −174.0 (4) | Ni1—N2—C12—C13 | 2.9 (5) |
| O1—Ni1—N3—C17 | −170.1 (3) | C17—N3—C13—C14 | 1.4 (6) |
| O3—Ni1—N3—C17 | 87.3 (4) | Ni1—N3—C13—C14 | −169.1 (4) |
| N6—Ni1—N3—C17 | −68.7 (4) | C17—N3—C13—C12 | 178.2 (4) |
| N5—Ni1—N3—C13 | 177.5 (3) | Ni1—N3—C13—C12 | 7.7 (4) |
| N2—Ni1—N3—C13 | −4.9 (3) | N2—C12—C13—N3 | −7.3 (6) |
| O1—Ni1—N3—C13 | −1.0 (5) | N2—C12—C13—C14 | 169.5 (4) |
| O3—Ni1—N3—C13 | −103.6 (3) | N3—C13—C14—C15 | −0.3 (7) |
| N6—Ni1—N3—C13 | 100.4 (3) | C12—C13—C14—C15 | −176.8 (4) |
| C18—N4—N5—C29 | 174.7 (4) | C13—C14—C15—C16 | −0.7 (8) |
| C18—N4—N5—Ni1 | −6.3 (4) | C14—C15—C16—C17 | 0.5 (8) |
| N2—Ni1—N5—C29 | 139 (2) | C13—N3—C17—C16 | −1.6 (6) |
| O1—Ni1—N5—C29 | 98.6 (3) | Ni1—N3—C17—C16 | 167.0 (3) |
| O3—Ni1—N5—C29 | −174.8 (3) | C15—C16—C17—N3 | 0.7 (7) |
| N6—Ni1—N5—C29 | 1.8 (3) | Ni1—O3—C18—N4 | 2.8 (5) |
| N3—Ni1—N5—C29 | −80.7 (3) | Ni1—O3—C18—C20 | −178.8 (2) |
| N2—Ni1—N5—N4 | −40 (2) | N5—N4—C18—O3 | 2.0 (5) |
| O1—Ni1—N5—N4 | −80.5 (3) | N5—N4—C18—C20 | −176.4 (3) |
| O3—Ni1—N5—N4 | 6.1 (2) | C24—C19—C20—C21 | 2.6 (6) |
| N6—Ni1—N5—N4 | −177.3 (3) | C24—C19—C20—C18 | −175.0 (3) |
| N3—Ni1—N5—N4 | 100.1 (3) | O3—C18—C20—C19 | 0.7 (5) |
| N5—Ni1—N6—C34 | −173.5 (4) | N4—C18—C20—C19 | 179.1 (3) |
| N2—Ni1—N6—C34 | 9.1 (4) | O3—C18—C20—C21 | −176.8 (4) |
| O1—Ni1—N6—C34 | 88.5 (4) | N4—C18—C20—C21 | 1.7 (5) |
| O3—Ni1—N6—C34 | −165.3 (3) | C19—C20—C21—O4 | 179.0 (4) |
| N3—Ni1—N6—C34 | −67.7 (4) | C18—C20—C21—O4 | −3.6 (6) |
| N5—Ni1—N6—C30 | −7.1 (3) | C19—C20—C21—C22 | −2.8 (6) |
| N2—Ni1—N6—C30 | 175.4 (3) | C18—C20—C21—C22 | 174.6 (4) |
| O1—Ni1—N6—C30 | −105.1 (3) | O4—C21—C22—C23 | 178.2 (4) |
| O3—Ni1—N6—C30 | 1.0 (5) | C20—C21—C22—C23 | 0.0 (6) |
| N3—Ni1—N6—C30 | 98.7 (3) | C21—C22—C23—C24 | 3.1 (6) |
| N5—Ni1—O1—C1 | 171.4 (3) | C21—C22—C23—C28 | −177.4 (4) |
| N2—Ni1—O1—C1 | −6.2 (3) | C20—C19—C24—C23 | 0.5 (6) |
| O3—Ni1—O1—C1 | 94.2 (3) | C20—C19—C24—C25 | 178.9 (4) |
| N6—Ni1—O1—C1 | −108.9 (3) | C22—C23—C24—C19 | −3.3 (6) |
| N3—Ni1—O1—C1 | −10.1 (5) | C28—C23—C24—C19 | 177.2 (4) |
| N5—Ni1—O3—C18 | −4.6 (2) | C22—C23—C24—C25 | 178.3 (4) |
| N2—Ni1—O3—C18 | 172.7 (2) | C28—C23—C24—C25 | −1.3 (6) |
| O1—Ni1—O3—C18 | 95.4 (2) | C19—C24—C25—C26 | −177.4 (4) |
| N6—Ni1—O3—C18 | −12.8 (4) | C23—C24—C25—C26 | 1.0 (6) |
| N3—Ni1—O3—C18 | −108.1 (2) | C24—C25—C26—C27 | 0.5 (7) |

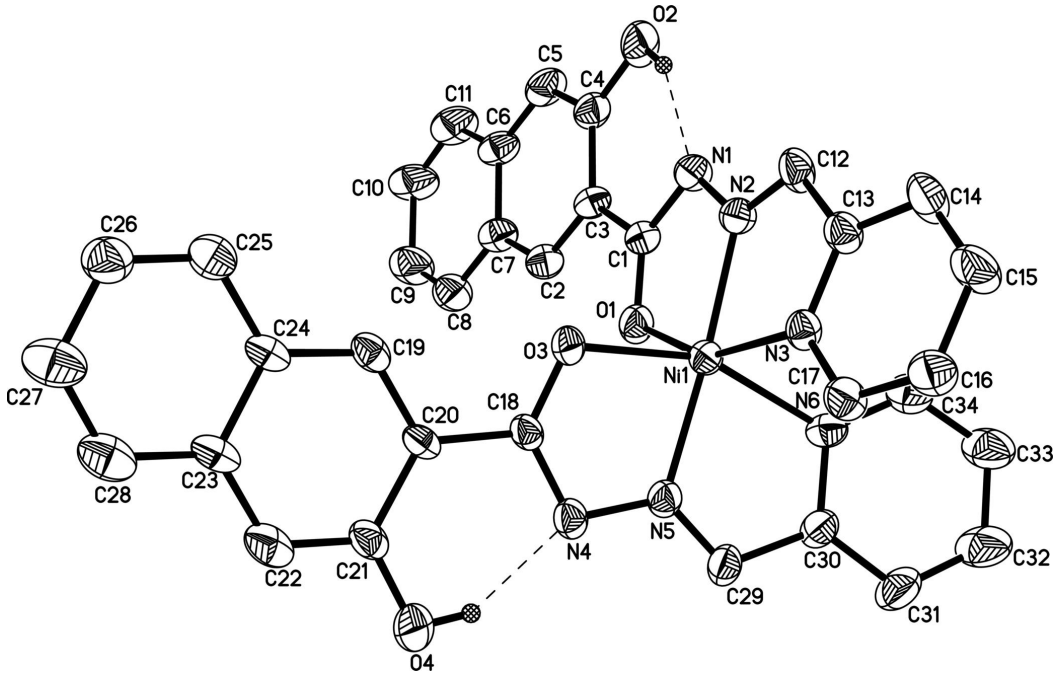
| | | | |
|---|---|---|---|
| Ni1—O1—C1—N1 | 6.8 (5) | C25—C26—C27—C28 | −1.9 (8) |
| Ni1—O1—C1—C3 | −173.8 (3) | C26—C27—C28—C23 | 1.6 (8) |
| N2—N1—C1—O1 | −2.3 (6) | C22—C23—C28—C27 | −179.5 (5) |
| N2—N1—C1—C3 | 178.3 (3) | C24—C23—C28—C27 | 0.0 (6) |
| C7—C2—C3—C4 | 0.1 (6) | N4—N5—C29—C30 | −177.3 (3) |
| C7—C2—C3—C1 | 178.1 (4) | Ni1—N5—C29—C30 | 3.7 (5) |
| O1—C1—C3—C2 | 0.0 (6) | C34—N6—C30—C31 | 1.8 (6) |
| N1—C1—C3—C2 | 179.4 (4) | Ni1—N6—C30—C31 | −166.2 (3) |
| O1—C1—C3—C4 | 177.9 (4) | C34—N6—C30—C29 | 179.1 (4) |
| N1—C1—C3—C4 | −2.7 (6) | Ni1—N6—C30—C29 | 11.1 (4) |
| C2—C3—C4—O2 | −179.5 (4) | N5—C29—C30—N6 | −10.3 (5) |
| C1—C3—C4—O2 | 2.6 (7) | N5—C29—C30—C31 | 167.0 (4) |
| C2—C3—C4—C5 | −0.7 (7) | N6—C30—C31—C32 | −0.3 (7) |
| C1—C3—C4—C5 | −178.7 (4) | C29—C30—C31—C32 | −177.4 (4) |
| O2—C4—C5—C6 | −179.8 (4) | C30—C31—C32—C33 | −1.3 (8) |
| C3—C4—C5—C6 | 1.3 (7) | C31—C32—C33—C34 | 1.5 (8) |
| C4—C5—C6—C7 | −1.2 (7) | C30—N6—C34—C33 | −1.6 (7) |
| C4—C5—C6—C11 | 178.7 (4) | Ni1—N6—C34—C33 | 163.8 (4) |
| C5—C6—C7—C8 | 179.6 (4) | C32—C33—C34—N6 | 0.0 (8) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H4···N4 | 0.82 | 1.83 | 2.561 (4) | 148 |
| O2—H2···N1 | 0.82 | 1.84 | 2.567 (4) | 147 |
| C15—H15···O4$^{i}$ | 0.93 | 2.56 | 3.195 (6) | 126 |
| C32—H32···O2$^{ii}$ | 0.93 | 2.38 | 3.295 (6) | 168 |

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*, *y*, *z*+1.

**Fig. 1**


O2
C5
C4
C11
C6
N1
C12
C10
N2
C13
C14
C26
C25
C3
C1
C15
C7
C2
C9
C8
O1
C24
C19
O3
N3
C27
Ni1
C17
C16
C18
N6
C34
C20
C33
C28
C23
N4
N5
C22
C21
C30
C32
C29
C31
O4