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cis-[(7*R*,14*R*)-5,5,7,12,12,14-Hexamethyl-1,4,8,11-tetraazacyclotetradecane- κ^4 N](oxalato- κ^2 O,O')nickel(II) oxalic acid solvate

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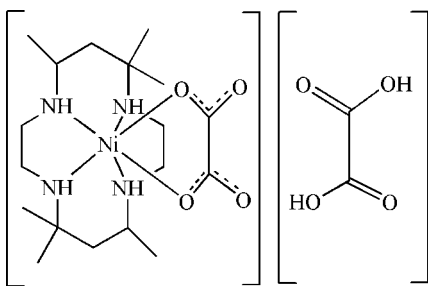
Received 14 May 2009; accepted 26 May 2009

 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.028; wR factor = 0.064; data-to-parameter ratio = 17.8.

Both molecules of the title compound, $[\text{Ni}(\text{C}_2\text{O}_4)(\text{C}_{16}\text{H}_{36}\text{N}_4)] \cdot \text{C}_2\text{H}_2\text{O}_4$, are located on a crystallographic twofold rotation axis. The Ni^{II} atom shows a distorted octahedral geometry. The crystal packing is stabilized by $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds.

Related literature

For general background, see: Tait & Busch (1976); Curtis (1965). For a related crystal structure, see: Tang *et al.* (2002).



Experimental

Crystal data

 $[\text{Ni}(\text{C}_2\text{O}_4)(\text{C}_{16}\text{H}_{36}\text{N}_4)] \cdot \text{C}_2\text{H}_2\text{O}_4$
 $M_r = 521.25$

 Orthorhombic, $P2_12_12$
 $a = 10.1261$ (15) Å

 $b = 15.515$ (2) Å
 $c = 8.0467$ (11) Å
 $V = 1264.2$ (3) Å³
 $Z = 2$

 Mo $K\alpha$ radiation

 $\mu = 0.82$ mm⁻¹
 $T = 173$ K

 $0.48 \times 0.21 \times 0.15$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Absorption correction: multi-scan

 (*SADABS*; Sheldrick, 1996)

 $T_{\text{min}} = 0.695$, $T_{\text{max}} = 0.887$

5665 measured reflections

2740 independent reflections

 2435 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.064$
 $S = 1.08$

2740 reflections

154 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.40$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Absolute structure: Flack (1983),

1131 Friedel pairs

Flack parameter: 0.027 (13)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|--|-------|--------------|--------------|----------------|
| $\text{N1}-\text{H1C} \cdots \text{O4}^{\text{i}}$ | 0.93 | 2.17 | 3.075 (2) | 164 |
| $\text{N2}-\text{H2C} \cdots \text{O2}^{\text{ii}}$ | 0.93 | 2.13 | 2.987 (2) | 152 |
| $\text{O3}-\text{H3A} \cdots \text{O2}^{\text{iii}}$ | 0.84 | 1.70 | 2.532 (2) | 170 |

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2961).

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supporting information

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***cis*-[*(7R,14R)*-5,5,7,12,12,14-Hexamethyl-1,4,8,11-tetraazacyclotetradecane- κ^4N](oxalato- κ^2O,O')nickel(II) oxalic acid solvate**

Guang-Chuan Ou, Yong-Qiang Dai and Man-Sheng Tang

S1. Comment

Recently, many helical structures have been constructed through the coordination interactions of the organic ligand with suitable metal ions. Helical polymers constructed *via* hydrogen bonding, which is a versatile and efficient strategy, are still rare, and only a few cases have been reported. Then we employ chiral macrocyclic ligand *L* and oxalic acid as building blocks to construct helical structure, and unfortunately the helical structure is not obtained.

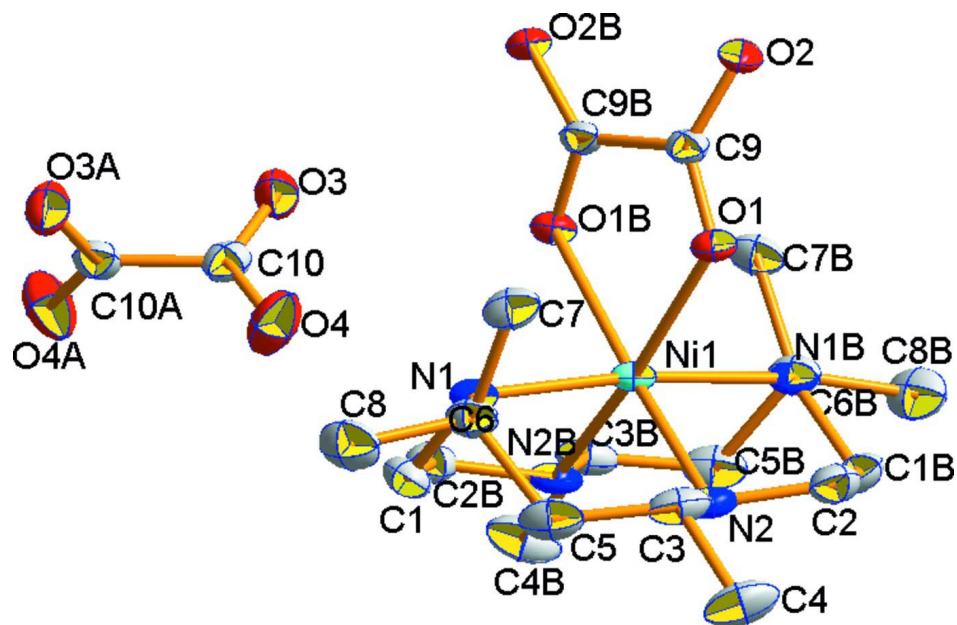
As illustrated in Fig.1, the six-coordinated Ni centre displays a distorted octahedral geometry. Neighbouring molecules are connected through intermolecular N-H \cdots O and O-H \cdots O hydrogen bonds (Fig. 2).

S2. Experimental

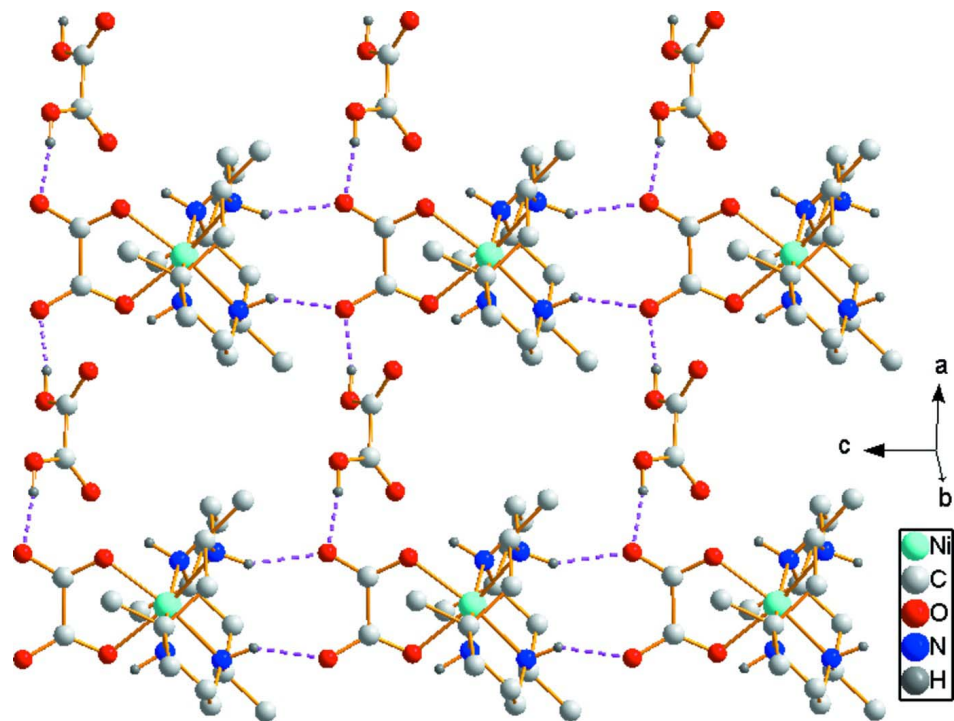
Oxalic acid (0.5 g, 4 mmol) and NaOH (0.08 g, 2 mmol) were dissolved in 15 ml of water. To this solution was added [Ni(C₁₆H₃₆N₄)](ClO₄)₂ (0.54 g, 1 mmol) dissolved in 2 ml of CH₃CN. The solution was left to stand at room temperature and violet crystals formed after several weeks.

S3. Refinement

All H atoms were placed in calculated positions (O—H = 0.84 Å, N—H = 0.93 Å, C—H 0.98 to 1.00 Å) and were included in the refinement in the riding model approximation, with U_{iso} (H) set to 1.2 U_{eq} (C,N) or 1.5 U_{eq} (C_{methyl},O).

**Figure 1**

The molecular structure of the title compound, showing displacement ellipsoids at the 50% probability level; symmetry codes for the generated atoms: A(1 - x, 2 - y, z), B(2 - x, -y, z). H-atoms have been excluded for clarity.

**Figure 2**

The hydrogen bond pattern in the title compound.

cis-[(7*R*,14*R*)-5,5,7,12,12,14-Hexamethyl-1,4,8,11-tetraazacyclotetradecane- κ^4N](oxalato- κ^2O,O')nickel(II)
oxalic acid solvate

Crystal data

[Ni(C₂O₄)(C₁₆H₃₆N₄)]·C₂H₂O₄
 $M_r = 521.25$
 Orthorhombic, $P2_12_12$
 Hall symbol: P 2 2ab
 $a = 10.1261$ (15) Å
 $b = 15.515$ (2) Å
 $c = 8.0467$ (11) Å
 $V = 1264.2$ (3) Å³
 $Z = 2$

$F(000) = 556$
 $D_x = 1.369$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 3630 reflections
 $\theta = 2.4$ – 26.9°
 $\mu = 0.82$ mm⁻¹
 $T = 173$ K
 Prism, violet
 $0.48 \times 0.21 \times 0.15$ mm

Data collection

Bruker SMART CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.695$, $T_{\max} = 0.887$

5665 measured reflections
 2740 independent reflections
 2435 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\max} = 27.1^\circ$, $\theta_{\min} = 2.4^\circ$
 $h = -12 \rightarrow 12$
 $k = -19 \rightarrow 8$
 $l = -10 \rightarrow 8$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.064$
 $S = 1.08$
 2740 reflections
 154 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0267P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.40$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³
 Absolute structure: Flack (1983), 1131 Friedel
 pairs
 Absolute structure parameter: 0.027 (13)

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 (Å²)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|-------------|----------------------------------|
| Ni1 | 1.0000 | 0.0000 | 0.60729 (3) | 0.02079 (9) |
| C10 | 0.57461 (18) | 1.00225 (19) | 0.2451 (2) | 0.0304 (4) |

| | | | | |
|-----|--------------|--------------|--------------|------------|
| O1 | 1.09484 (13) | 0.05954 (8) | 0.40383 (17) | 0.0245 (3) |
| N1 | 0.81963 (17) | 0.07108 (10) | 0.6220 (2) | 0.0256 (4) |
| H1C | 0.7759 | 0.0597 | 0.5227 | 0.031* |
| C3 | 1.0605 (3) | 0.17864 (15) | 0.7602 (3) | 0.0338 (6) |
| H3 | 1.0887 | 0.1991 | 0.6478 | 0.041* |
| C5 | 0.9146 (3) | 0.19508 (15) | 0.7775 (3) | 0.0366 (6) |
| H5A | 0.9025 | 0.2577 | 0.7950 | 0.044* |
| H5B | 0.8846 | 0.1659 | 0.8801 | 0.044* |
| N2 | 1.09305 (18) | 0.08487 (10) | 0.7733 (2) | 0.0271 (4) |
| H2C | 1.0718 | 0.0672 | 0.8804 | 0.032* |
| O2 | 1.12362 (14) | 0.03860 (10) | 0.13153 (16) | 0.0360 (4) |
| O3 | 0.62920 (15) | 0.94107 (11) | 0.1583 (2) | 0.0423 (4) |
| H3A | 0.7117 | 0.9467 | 0.1609 | 0.064* |
| O4 | 0.63195 (17) | 1.05745 (11) | 0.3227 (2) | 0.0520 (5) |
| C2 | 1.2350 (2) | 0.06808 (14) | 0.7481 (3) | 0.0331 (5) |
| H2A | 1.2869 | 0.0964 | 0.8369 | 0.040* |
| H2B | 1.2636 | 0.0920 | 0.6399 | 0.040* |
| C9 | 1.0624 (2) | 0.02855 (12) | 0.2656 (2) | 0.0249 (4) |
| C1 | 0.7410 (2) | 0.02740 (14) | 0.7515 (3) | 0.0334 (5) |
| H1A | 0.6460 | 0.0390 | 0.7329 | 0.040* |
| H1B | 0.7652 | 0.0505 | 0.8620 | 0.040* |
| C6 | 0.8215 (2) | 0.16770 (13) | 0.6365 (2) | 0.0301 (5) |
| C4 | 1.1353 (3) | 0.23189 (14) | 0.8912 (3) | 0.0486 (7) |
| H4A | 1.1131 | 0.2930 | 0.8782 | 0.073* |
| H4B | 1.2306 | 0.2240 | 0.8764 | 0.073* |
| H4C | 1.1097 | 0.2126 | 1.0027 | 0.073* |
| C8 | 0.6827 (3) | 0.20270 (16) | 0.6697 (3) | 0.0452 (6) |
| H8A | 0.6209 | 0.1782 | 0.5888 | 0.068* |
| H8B | 0.6833 | 0.2656 | 0.6594 | 0.068* |
| H8C | 0.6551 | 0.1866 | 0.7823 | 0.068* |
| C7 | 0.8682 (2) | 0.20360 (14) | 0.4693 (3) | 0.0343 (5) |
| H7A | 0.9557 | 0.1803 | 0.4432 | 0.051* |
| H7B | 0.8731 | 0.2666 | 0.4756 | 0.051* |
| H7C | 0.8057 | 0.1869 | 0.3821 | 0.051* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|---------------|--------------|--------------|
| Ni1 | 0.02928 (17) | 0.02073 (15) | 0.01236 (14) | -0.00015 (18) | 0.000 | 0.000 |
| C10 | 0.0352 (10) | 0.0320 (10) | 0.0240 (9) | -0.0018 (13) | -0.0030 (8) | 0.0069 (14) |
| O1 | 0.0311 (7) | 0.0263 (7) | 0.0160 (7) | 0.0006 (6) | -0.0020 (6) | 0.0021 (6) |
| N1 | 0.0341 (9) | 0.0269 (8) | 0.0159 (8) | 0.0017 (7) | 0.0010 (8) | -0.0023 (7) |
| C3 | 0.0562 (15) | 0.0247 (11) | 0.0205 (11) | -0.0054 (11) | -0.0064 (11) | -0.0006 (9) |
| C5 | 0.0602 (18) | 0.0221 (11) | 0.0277 (13) | 0.0046 (11) | -0.0021 (12) | -0.0058 (10) |
| N2 | 0.0421 (11) | 0.0249 (9) | 0.0142 (8) | -0.0023 (8) | -0.0032 (7) | 0.0006 (7) |
| O2 | 0.0294 (8) | 0.0628 (10) | 0.0158 (7) | -0.0010 (7) | 0.0028 (6) | 0.0070 (7) |
| O3 | 0.0281 (8) | 0.0581 (11) | 0.0408 (9) | -0.0047 (8) | 0.0026 (7) | -0.0161 (8) |
| O4 | 0.0439 (10) | 0.0474 (10) | 0.0647 (12) | 0.0033 (8) | -0.0190 (9) | -0.0187 (9) |

| | | | | | | |
|----|-------------|-------------|-------------|--------------|--------------|--------------|
| C2 | 0.0385 (13) | 0.0368 (13) | 0.0239 (11) | -0.0086 (11) | -0.0034 (10) | -0.0029 (10) |
| C9 | 0.0278 (10) | 0.0297 (10) | 0.0170 (9) | 0.0073 (8) | -0.0008 (8) | 0.0048 (8) |
| C1 | 0.0356 (12) | 0.0391 (13) | 0.0255 (11) | 0.0041 (10) | 0.0104 (10) | -0.0003 (9) |
| C6 | 0.0404 (13) | 0.0262 (10) | 0.0238 (11) | 0.0077 (9) | -0.0002 (10) | -0.0034 (9) |
| C4 | 0.0816 (19) | 0.0296 (11) | 0.0346 (13) | -0.0077 (12) | -0.0185 (15) | -0.0059 (12) |
| C8 | 0.0556 (16) | 0.0412 (13) | 0.0388 (13) | 0.0170 (12) | 0.0040 (12) | -0.0066 (11) |
| C7 | 0.0477 (14) | 0.0283 (11) | 0.0269 (11) | 0.0057 (10) | -0.0066 (11) | 0.0049 (9) |

Geometric parameters (Å, °)

| | | | |
|--------------------------------------|-------------|-------------------------|-------------|
| Ni1—N2 | 2.0990 (17) | N2—H2C | 0.9300 |
| Ni1—N2 ⁱ | 2.0990 (17) | O2—C9 | 1.254 (2) |
| Ni1—O1 ⁱ | 2.1110 (13) | O3—H3A | 0.8400 |
| Ni1—O1 | 2.1110 (13) | C2—C1 ⁱ | 1.501 (3) |
| Ni1—N1 | 2.1368 (16) | C2—H2A | 0.9900 |
| Ni1—N1 ⁱ | 2.1368 (16) | C2—H2B | 0.9900 |
| C10—O4 | 1.209 (3) | C9—C9 ⁱ | 1.543 (4) |
| C10—O3 | 1.302 (3) | C1—C2 ⁱ | 1.501 (3) |
| C10—C10 ⁱⁱ | 1.513 (4) | C1—H1A | 0.9900 |
| O1—C9 | 1.255 (2) | C1—H1B | 0.9900 |
| N1—C1 | 1.477 (3) | C6—C8 | 1.530 (3) |
| N1—C6 | 1.504 (2) | C6—C7 | 1.531 (3) |
| N1—H1C | 0.9300 | C4—H4A | 0.9800 |
| C3—N2 | 1.495 (3) | C4—H4B | 0.9800 |
| C3—C5 | 1.506 (3) | C4—H4C | 0.9800 |
| C3—C4 | 1.539 (3) | C8—H8A | 0.9800 |
| C3—H3 | 1.0000 | C8—H8B | 0.9800 |
| C5—C6 | 1.535 (3) | C8—H8C | 0.9800 |
| C5—H5A | 0.9900 | C7—H7A | 0.9800 |
| C5—H5B | 0.9900 | C7—H7B | 0.9800 |
| N2—C2 | 1.475 (3) | C7—H7C | 0.9800 |
| N2—Ni1—N2 ⁱ | 100.97 (9) | Ni1—N2—H2C | 107.5 |
| N2—Ni1—O1 ⁱ | 166.48 (6) | C10—O3—H3A | 109.5 |
| N2 ⁱ —Ni1—O1 ⁱ | 90.84 (6) | N2—C2—C1 ⁱ | 109.25 (19) |
| N2—Ni1—O1 | 90.84 (6) | N2—C2—H2A | 109.8 |
| N2 ⁱ —Ni1—O1 | 166.48 (6) | C1 ⁱ —C2—H2A | 109.8 |
| O1 ⁱ —Ni1—O1 | 78.29 (7) | N2—C2—H2B | 109.8 |
| N2—Ni1—N1 | 91.42 (7) | C1 ⁱ —C2—H2B | 109.8 |
| N2 ⁱ —Ni1—N1 | 84.55 (6) | H2A—C2—H2B | 108.3 |
| O1 ⁱ —Ni1—N1 | 83.10 (6) | O2—C9—O1 | 125.80 (19) |
| O1—Ni1—N1 | 101.88 (5) | O2—C9—C9 ⁱ | 118.43 (12) |
| N2—Ni1—N1 ⁱ | 84.55 (6) | O1—C9—C9 ⁱ | 115.74 (11) |
| N2 ⁱ —Ni1—N1 ⁱ | 91.42 (7) | N1—C1—C2 ⁱ | 110.65 (19) |
| O1 ⁱ —Ni1—N1 ⁱ | 101.88 (5) | N1—C1—H1A | 109.5 |
| O1—Ni1—N1 ⁱ | 83.10 (6) | C2 ⁱ —C1—H1A | 109.5 |
| N1—Ni1—N1 ⁱ | 173.67 (9) | N1—C1—H1B | 109.5 |
| O4—C10—O3 | 126.15 (19) | C2 ⁱ —C1—H1B | 109.5 |

| | | | |
|----------------------------|--------------|----------------------------|--------------|
| O4—C10—C10 ⁱⁱ | 120.8 (3) | H1A—C1—H1B | 108.1 |
| O3—C10—C10 ⁱⁱ | 113.0 (3) | N1—C6—C8 | 110.86 (18) |
| C9—O1—Ni1 | 113.59 (12) | N1—C6—C7 | 107.34 (15) |
| C1—N1—C6 | 114.16 (15) | C8—C6—C7 | 107.94 (18) |
| C1—N1—Ni1 | 105.25 (12) | N1—C6—C5 | 109.95 (17) |
| C6—N1—Ni1 | 120.54 (13) | C8—C6—C5 | 109.69 (18) |
| C1—N1—H1C | 105.2 | C7—C6—C5 | 111.02 (19) |
| C6—N1—H1C | 105.2 | C3—C4—H4A | 109.5 |
| Ni1—N1—H1C | 105.2 | C3—C4—H4B | 109.5 |
| N2—C3—C5 | 112.0 (2) | H4A—C4—H4B | 109.5 |
| N2—C3—C4 | 111.46 (19) | C3—C4—H4C | 109.5 |
| C5—C3—C4 | 109.2 (2) | H4A—C4—H4C | 109.5 |
| N2—C3—H3 | 108.0 | H4B—C4—H4C | 109.5 |
| C5—C3—H3 | 108.0 | C6—C8—H8A | 109.5 |
| C4—C3—H3 | 108.0 | C6—C8—H8B | 109.5 |
| C3—C5—C6 | 119.2 (2) | H8A—C8—H8B | 109.5 |
| C3—C5—H5A | 107.5 | C6—C8—H8C | 109.5 |
| C6—C5—H5A | 107.5 | H8A—C8—H8C | 109.5 |
| C3—C5—H5B | 107.5 | H8B—C8—H8C | 109.5 |
| C6—C5—H5B | 107.5 | C6—C7—H7A | 109.5 |
| H5A—C5—H5B | 107.0 | C6—C7—H7B | 109.5 |
| C2—N2—C3 | 112.13 (17) | H7A—C7—H7B | 109.5 |
| C2—N2—Ni1 | 103.84 (12) | C6—C7—H7C | 109.5 |
| C3—N2—Ni1 | 117.82 (13) | H7A—C7—H7C | 109.5 |
| C2—N2—H2C | 107.5 | H7B—C7—H7C | 109.5 |
| C3—N2—H2C | 107.5 | | |
| N2—Ni1—O1—C9 | -177.81 (13) | O1—Ni1—N2—C2 | -60.63 (12) |
| N2 ⁱ —Ni1—O1—C9 | 31.2 (3) | N1—Ni1—N2—C2 | -162.53 (13) |
| O1 ⁱ —Ni1—O1—C9 | -5.92 (10) | N1 ⁱ —Ni1—N2—C2 | 22.36 (12) |
| N1—Ni1—O1—C9 | -86.19 (13) | N2 ⁱ —Ni1—N2—C3 | -122.59 (18) |
| N1 ⁱ —Ni1—O1—C9 | 97.78 (13) | O1 ⁱ —Ni1—N2—C3 | 27.8 (4) |
| N2—Ni1—N1—C1 | -94.43 (13) | O1—Ni1—N2—C3 | 64.03 (16) |
| N2 ⁱ —Ni1—N1—C1 | 6.46 (13) | N1—Ni1—N2—C3 | -37.87 (16) |
| O1 ⁱ —Ni1—N1—C1 | 97.97 (13) | N1 ⁱ —Ni1—N2—C3 | 147.02 (16) |
| O1—Ni1—N1—C1 | 174.42 (12) | C3—N2—C2—C1 ⁱ | -176.24 (19) |
| N1 ⁱ —Ni1—N1—C1 | -44.13 (12) | Ni1—N2—C2—C1 ⁱ | -48.0 (2) |
| N2—Ni1—N1—C6 | 36.38 (14) | Ni1—O1—C9—O2 | -163.44 (16) |
| N2 ⁱ —Ni1—N1—C6 | 137.26 (14) | Ni1—O1—C9—C9 ⁱ | 14.9 (2) |
| O1 ⁱ —Ni1—N1—C6 | -131.22 (14) | C6—N1—C1—C2 ⁱ | -169.19 (19) |
| O1—Ni1—N1—C6 | -54.78 (14) | Ni1—N1—C1—C2 ⁱ | -34.8 (2) |
| N1 ⁱ —Ni1—N1—C6 | 86.67 (13) | C1—N1—C6—C8 | -45.1 (2) |
| N2—C3—C5—C6 | -70.9 (3) | Ni1—N1—C6—C8 | -171.97 (14) |
| C4—C3—C5—C6 | 165.16 (19) | C1—N1—C6—C7 | -162.80 (18) |
| C5—C3—N2—C2 | 177.2 (2) | Ni1—N1—C6—C7 | 70.36 (19) |
| C4—C3—N2—C2 | -60.2 (2) | C1—N1—C6—C5 | 76.3 (2) |
| C5—C3—N2—Ni1 | 56.7 (2) | Ni1—N1—C6—C5 | -50.5 (2) |
| C4—C3—N2—Ni1 | 179.33 (16) | C3—C5—C6—N1 | 66.3 (3) |

| | | | |
|----------------------------|-------------|-------------|------------|
| N2 ⁱ —Ni1—N2—C2 | 112.75 (13) | C3—C5—C6—C8 | -171.6 (2) |
| O1 ⁱ —Ni1—N2—C2 | -96.8 (3) | C3—C5—C6—C7 | -52.4 (3) |

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

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N1—H1C \cdots O4 ⁱⁱⁱ | 0.93 | 2.17 | 3.075 (2) | 164 |
| N2—H2C \cdots O2 ^{iv} | 0.93 | 2.13 | 2.987 (2) | 152 |
| O3—H3A \cdots O2 ^v | 0.84 | 1.70 | 2.532 (2) | 170 |

Symmetry codes: (iii) $x, y-1, z$; (iv) $x, y, z+1$; (v) $-x+2, -y+1, z$.