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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.034
wR factor = 0.093
Data-to-parameter ratio = 26.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Bis[1-(2,6-dimethylanilino)propane-1,2-dione dioximato]nickel(II)

The structure of the title complex consists of isolated $[\text{Ni}(\text{C}_{11}\text{H}_{14}\text{N}_3\text{O}_2)_2]$ units. The Ni atom is coordinated by four oxime N atoms in distorted square-planar geometry and lies on an inversion centre. The structure is stabilized by strong intramolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds and a possible $\text{N}-\text{H}\cdots\pi$ intermolecular interaction.

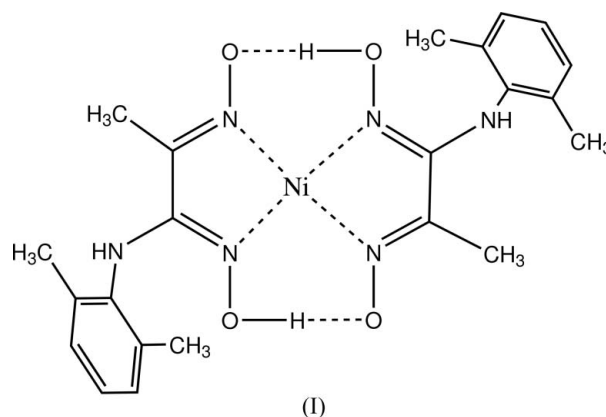
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Comment

This work is part of our ongoing research on the synthesis and characterization of new *vic*-dioximes and their transition metal complexes (Zülfikarođlu *et al.*, 2003). Metal complexes of various glyoximate ligands have long been of importance in analytical chemistry and medicine (Chakravorty, 1974; Michael *et al.*, 2000).



In the title compound, (I), alternately named bis[*N*-(2,6-dimethylphenyl)aminomethylglyoximato-*N,N'*]nickel(II) (Fig. 1), the Ni atom, which lies on a site of $\bar{1}$ symmetry, is coordinated by four oxime N atoms arising from two bidentate ligand molecules. The local coordination of the NiN_4 chromophore is distorted square planar (D_{2h} symmetry). The Ni–N and $\text{O1}\cdots\text{O2}^i$ distances (Tables 1 and 2; symmetry code as in these tables) are similar to the distances found in the related complexes bis[*N*-(2,6-dimethylphenyl)aminoglyoximato-*N,N'*]nickel(II) (Ülkü *et al.*, 1996), bis[*N*-(4-methylphenyl)aminoglyoximato-*N,N'*]nickel(II) (Isik *et al.*, 2000), bis[*N*-(2,6-dimethylphenyl)aminophenylglyoximato- κ^2N,N']nickel(II) dimethyl sulfoxide solvate (Batı *et al.*, 2004) and bis[*N*-(4-methoxyphenyl)aminomethylglyoximato]nickel(II) (Batı *et al.*, 2005). In these, one Ni–N bond is significantly longer than the other (by between 0.02 and 0.05 Å). This difference can possibly be attributed to the different groups attached to oxime atoms C9 and C10.

The different N–O bond lengths reflect the chemically distinct O atoms. The oxime group has an *E* configuration with planar O1–N2–C9–C10. The oxime OH group is adjacent to the bridging amine group in all complexes, and in (I) accepts an intraligand N–H···O bond. The benzene and five-membered chelate (NiC₂N₂) rings in (I) are essentially planar, with r.m.s. deviations of only 0.0045 and 0.0159 Å.

Comparison of the bond lengths of the oxime group with those of the free ligand (Hökelek *et al.*, 2001) reveals that, upon complex formation, the N2–O1, N3–O2 and C9–C10 distances are shortened by 0.040, 0.078 and 0.018 Å, respectively, whereas the C9–N2 and C10–N3 distances are increased by 0.013 and 0.028 Å, respectively.

The intramolecular inter-ligand O···O separations in these compounds are all similar, lying between 2.462 (3) and 2.547 (3) Å. Such short O···O separations are often associated with symmetrical O···H···O hydrogen bonds (Chakravorty, 1974). In (I), one of the O-bound acidic H atoms is lost from each ligand during complex formation and the remaining O-bound H atom participates in a very strong intramolecular hydrogen bond to the adjacent O atom (Table 2). The H atom was clearly visible in a difference map and, like the other complexes noted above, the O–H···O bond is not symmetrical.

An analysis of the intermolecular contacts in (I) with *PLATON* (Spek, 2003) revealed a possible weak N–H···π(–*x*, 1 – *y*, 2 – *z*) interaction between the amine H atom and an adjacent benzene ring (atoms C1–C6) with an H···π distance of 2.965 (16) Å.

Experimental

1-(2,6-Dimethylphenylamino)propane-1,2-dione dioxime (*L*) was prepared according to the method of Hökelek *et al.* (2001). A solution of NiCl₂·6H₂O (0.48 g, 2 mmol) in ethanol–water (1:1) was added dropwise to a solution of *L* (0.882 g, 4 mmol) in ethanol (20 ml). A 1% solution of KOH in water was then dripped slowly into the mixture until the pH reached 5.5. The resulting precipitate was removed by suction filtration, washed and dried *in vacuo*. Recrystallization from a chloroform–ethanol mixture (2:1) gave orange rod crystals of (I).

Crystal data

[Ni(C ₁₁ H ₁₄ N ₃ O ₂) ₂]	<i>D_x</i> = 1.433 Mg m ^{−3}
<i>M_r</i> = 499.21	Mo <i>K</i> α radiation
Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Cell parameters from 4650 reflections
<i>a</i> = 8.1081 (4) Å	<i>θ</i> = 2.5–32.0°
<i>b</i> = 16.0311 (8) Å	<i>μ</i> = 0.88 mm ^{−1}
<i>c</i> = 8.9223 (4) Å	<i>T</i> = 293 (2) K
<i>β</i> = 94.202 (1)°	Rod, orange
<i>V</i> = 1156.62 (10) Å ³	0.49 × 0.30 × 0.24 mm
<i>Z</i> = 2	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	4184 independent reflections
<i>ω</i> scans	2988 reflections with <i>I</i> > 2σ(<i>I</i>)
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1997)	<i>R</i> _{int} = 0.021
<i>T</i> _{min} = 0.736, <i>T</i> _{max} = 0.810	<i>θ</i> _{max} = 32.5°
11778 measured reflections	<i>h</i> = −12 → 11
	<i>k</i> = −24 → 16
	<i>l</i> = −13 → 13

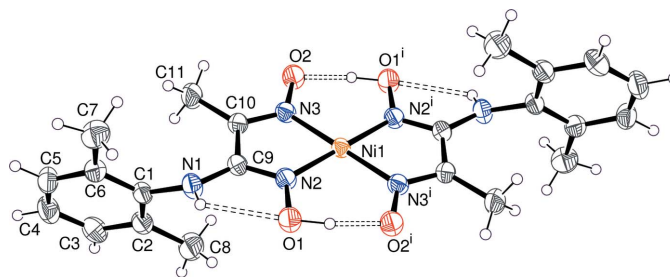


Figure 1
View of (I) showing 40% probability displacement ellipsoids (arbitrary spheres for the H atoms) and hydrogen bonds as dashed lines. [Symmetry code: (i) 1 – *x*, 1 – *y*, 1 – *z*.]

Refinement

Refinement on <i>F</i> ²	H atoms treated by a mixture of independent and constrained refinement
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.034	<i>w</i> = 1/[σ ² (<i>F</i> _o ²) + (0.0541 <i>P</i>) ²]
<i>wR</i> (<i>F</i> ²) = 0.093	where <i>P</i> = (<i>F</i> _o ² + 2 <i>F</i> _c ²)/3
<i>S</i> = 0.98	(Δ/σ) _{max} < 0.001
4184 reflections	Δρ _{max} = 0.45 e Å ^{−3}
159 parameters	Δρ _{min} = −0.16 e Å ^{−3}

Table 1

Selected geometric parameters (Å, °).

Ni1–N2	1.8397 (11)	Ni1–N3	1.8779 (11)
N2–Ni1–N3 ⁱ	97.62 (5)	N2–Ni1–N3	82.38 (5)

Symmetry code: (i) 1 – *x*, 1 – *y*, 1 – *z*.

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O1–H1O···O2 ⁱ	0.86 (1)	1.65 (1)	2.4972 (14)	171 (2)
N1–H1N···O1	0.82 (1)	2.20 (2)	2.6361 (16)	113 (1)

Symmetry code: (i) 1 – *x*, 1 – *y*, 1 – *z*.

The O- and N-bound H atoms were found in difference maps and were refined with distance restraints [O–H = 0.84 (2) Å and N–H = 0.86 (2) Å] and with *U*_{iso}(H) = 1.2*U*_{eq}(carrier). C-bound H atoms were placed in calculated positions (C–H = 0.93–0.96 Å) and refined as riding, with *U*_{iso}(H) = 1.2 *U*_{eq}(carrier) or *U*_{iso}(H) = 1.5*U*_{eq}(methyl carrier). The –CH₃ groups were rotated to fit the electron density.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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References

- Bati, H., Dağ, C., Soylu, M. S., Taş, M., Çalışkan, N. & Büyüküngör, O. (2005). *Acta Cryst.* **E61**, m1866–m1868.

- Bati, H., ZülfiKaroglu, A., Taş, M., Çalışkan, N., Soylu, S., Andac, O. & Büyükgüngör, O. (2004). *Acta Cryst.* **E60**, m1334–m1336.
- Bruker (1999). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chakravorty, A. (1974). *Coord. Chem. Rev.* **13**, 1–46.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Hökelek, T., ZülfiKaroglu, A. & Bati, H. (2001). *Acta Cryst.* **E57**, o1247–o1249.
- Isik, S., Ozturk, S., Erdonmez, A., Macit, M. & Fun, H.-K. (2000). *Anal. Sci.* **16**, 559–560.
- Michael, J. P., Anthony, W. A. & Raymond, J. B. (2000). *Inorg. Chim. Acta*, **300–302**, 992–1003.
- Sheldrick, G. M. (1997). *SADABS*, *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Ülkü, D., Ercan, F., Macit, M. & Gulce, A. (1996). *Acta Cryst.* **C52**, 2680–2682.
- ZülfiKaroglu, A., Taş, M., Bati, H. & Bati, B. (2003). *Synth. React. Inorg. Met.-Org. Chem.* **33**, 625–638.

supporting information

Acta Cryst. (2005). E61, m2033–m2035 [doi:10.1107/S1600536805028965]

Bis[1-(2,6-dimethylanilino)propane-1,2-dione dioximato]nickel(II)**Hümeýra Batı, Ayşin Zülfikaroğlu, Murat Taş, Omer Andac and William T. A. Harrison****S1. Comment**

This work is part of our ongoing research on the synthesis and characterization of new vic dioximes and their transition metal complexes (Zülfikaroglu *et al.*, 2003). Metal complexes of various glyoximate ligands have long been of importance in analytical chemistry and medicine (Chakravorty, 1974; Michael *et al.*, 2000). In the title compound, (I), alternately named bis[*N*-(2,6-dimethylphenyl)aminomethylglyoximato-*N,N'*]nickel(II) (Fig. 1), the Ni atom that lies on a site of -1 symmetry, is coordinated by four oxime N atoms arising from two bidentate ligand molecules. The local coordination of the NiN₄ chromophore is distorted square planar (D_{2h} symmetry). The Ni—N and O1 \cdots O2ⁱ distances (Tables 1 and 2) are similar to the distances found in the related complexes bis[*N*-(2,6-dimethylphenyl)aminoglyoximato-*N,N'*]nickel(II) (Ulku *et al.*, 1996), bis[*N*-(4-methylphenyl)aminoglyoximato-*N,N'*]nickel(II) (Isik *et al.*, 2000), bis[*N*-(2,6-dimethylphenyl)aminophenylglyoximato- κ^2N,N']nickel(II) dimethyl sulfoxide solvate (Batı *et al.*, 2004) and bis[*N*-(4-methoxyphenyl)aminomethylglyoximato]nickel(II) (Batı *et al.*, 2005). In these, one Ni—N bond is significantly longer than the other (by between 0.02 and 0.05 Å). This difference can possibly be attributed to the different groups attached to oxime atoms C9 and C10. The intramolecular, inter-ligand, O \cdots O separations in these compounds are all similar, lying between 2.462 (3) and 2.547 (3) Å. Such short O \cdots O separations are often associated with symmetrical O \cdots H \cdots O hydrogen bonds (Chakravorty, 1974). In (I), one of the O-bound acidic H atoms is lost from each ligand during complex formation and the remaining O-bound H atom participates in a very strong intramolecular hydrogen bond to the adjacent O atom (Table 2). The H atom was clearly visible in a difference map and, like the other complexes noted above, the O—H \cdots O bond is not symmetrical. The different N—O bond lengths reflect the chemically distinct O atoms. The oxime group has an *E* configuration with planar O1—N2—C9—C10. The oxime —OH group is adjacent to the bridging amine group in all complexes, and in (I) accepts an intraligand N—H \cdots O bond. The phenyl and five-membered chelate (NiC₂N₂) rings in (I) are almost planar, with r.m.s deviations of 0.0045 and 0.0159 Å only.

Comparison of the bond lengths of the oxime group with those of the free ligand (Hökelek *et al.*, 2001) reveals that, upon complex formation, the N2—O1, N3—O3 and C9—C10 distances are shortened by 0.040, 0.078 and 0.018 Å, whereas the C9—N2 and C10—N3 distances are increased by 0.013 and 0.028 Å.

An analysis of the inter-molecular contacts in (I) with *PLATON* (Spek, 2003) reveals a possible weak N—H \cdots $\pi(-x, 1 - y, 2 - z)$ interaction between the amine H atom and an adjacent phenyl ring (atoms C1—C6) with an H \cdots π distance of 2.965 (16) Å.

S2. Experimental

1-(2,6-Dimethylphenylamino)propane-1,2-dione dioxime (*L*) was prepared according to the method of Hökelek *et al.* (2001). A solution of NiCl₂·6H₂O (0.48 g, 2 mmol) in ethanol–water (1:1) was added dropwise to a solution of *L* (0.882 g, 4 mmol) in ethanol (20 ml). Then, a 1% solution of KOH in water was dripped slowly into the mixture until the pH reached 5.5. The resulting precipitate was removed by suction filtration, washed and dried *in vacuo*. Recrystallization

from a chloroform–ethanol mixture (2:1) gave orange rod crystals of (I).

S3. Refinement

The O- and N-bound H atoms were found in difference maps and were refined with distance restraints [$\text{O—H} = 0.84$ (2) Å and $\text{N—H} = 0.86$ (2) Å] and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ to yield the final values given in Table 2. C-bound H atoms were placed in calculated positions ($\text{C—H} = 0.93\text{--}0.96$ Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl carrier})$. The $-\text{CH}_3$ groups were rotated to fit the electron density.

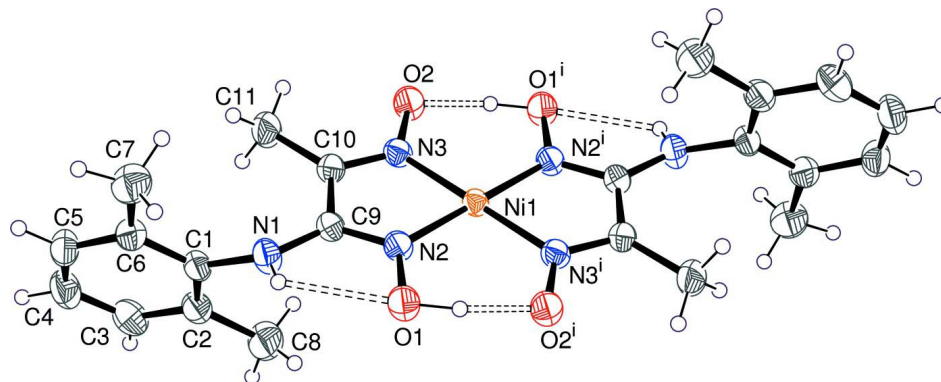


Figure 1

View of (I) showing 40% probability displacement ellipsoids (arbitrary spheres for the H atoms) and hydrogen bonds as dashed lines. [Symmetry code: (i) $1 - x, 1 - y, 1 - z$.]

Bis[1-(2,6-dimethylanilino)propane-1,2-dione dioximato]nickel(II)

Crystal data

$[\text{Ni}(\text{C}_{11}\text{H}_{14}\text{N}_3\text{O}_2)_2]$

$M_r = 499.21$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 8.1081$ (4) Å

$b = 16.0311$ (8) Å

$c = 8.9223$ (4) Å

$\beta = 94.202$ (1) $^\circ$

$V = 1156.62$ (10) Å³

$Z = 2$

$F(000) = 524$

$D_x = 1.433$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4650 reflections

$\theta = 2.5\text{--}32.0^\circ$

$\mu = 0.88$ mm⁻¹

$T = 293$ K

Rod, orange

$0.49 \times 0.30 \times 0.24$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1997)

$T_{\text{min}} = 0.736$, $T_{\text{max}} = 0.810$

11778 measured reflections

4184 independent reflections

2988 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\text{max}} = 32.5^\circ$, $\theta_{\text{min}} = 2.5^\circ$

$h = -12 \rightarrow 11$

$k = -24 \rightarrow 16$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.093$

$S = 0.98$

4184 reflections

159 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: none
 Hydrogen site location: difmap (O-H and N-H)
 and geom (C-H)

H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0541P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.5000	0.5000	0.5000	0.03193 (8)
O1	0.31671 (14)	0.39481 (6)	0.69080 (12)	0.0479 (2)
H1O	0.361 (2)	0.3665 (12)	0.6227 (19)	0.082 (7)*
O2	0.55255 (14)	0.67355 (6)	0.52231 (12)	0.0491 (2)
N1	0.23048 (16)	0.52210 (8)	0.85500 (15)	0.0444 (3)
H1N	0.221 (2)	0.4716 (9)	0.8697 (19)	0.047 (5)*
N2	0.37428 (14)	0.47315 (8)	0.65657 (13)	0.0379 (2)
N3	0.47882 (13)	0.60817 (7)	0.57706 (13)	0.0373 (2)
C1	0.11259 (16)	0.58122 (9)	0.90465 (15)	0.0391 (3)
C2	-0.01565 (17)	0.60907 (10)	0.80574 (16)	0.0460 (3)
C3	-0.13125 (19)	0.66334 (11)	0.86028 (19)	0.0550 (4)
H3	-0.2169	0.6837	0.7955	0.066*
C4	-0.1203 (2)	0.68714 (11)	1.0086 (2)	0.0582 (4)
H4	-0.1994	0.7226	1.0441	0.070*
C5	0.0076 (2)	0.65839 (11)	1.10458 (18)	0.0532 (4)
H5	0.0139	0.6750	1.2047	0.064*
C6	0.12720 (17)	0.60536 (10)	1.05560 (16)	0.0439 (3)
C7	0.2701 (2)	0.57673 (12)	1.15995 (18)	0.0615 (4)
H7A	0.2558	0.5965	1.2597	0.092*
H7B	0.3714	0.5985	1.1264	0.092*
H7C	0.2744	0.5169	1.1603	0.092*
C8	-0.0333 (2)	0.58167 (14)	0.64311 (18)	0.0672 (5)
H8A	0.0450	0.6113	0.5874	0.101*
H8B	-0.1434	0.5935	0.6015	0.101*
H8C	-0.0126	0.5228	0.6372	0.101*
C9	0.32550 (16)	0.53464 (9)	0.73794 (15)	0.0369 (3)
C10	0.39180 (16)	0.61579 (9)	0.69449 (15)	0.0388 (3)
C11	0.3760 (2)	0.69658 (10)	0.7739 (2)	0.0606 (4)
H11A	0.4560	0.7353	0.7409	0.091*

H11B	0.3949	0.6880	0.8803	0.091*
H11C	0.2668	0.7186	0.7518	0.091*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02994 (11)	0.03035 (12)	0.03578 (12)	0.00224 (8)	0.00434 (8)	-0.00006 (9)
O1	0.0569 (6)	0.0344 (5)	0.0547 (6)	-0.0031 (4)	0.0195 (5)	0.0013 (5)
O2	0.0568 (6)	0.0315 (5)	0.0612 (6)	-0.0033 (4)	0.0193 (5)	0.0003 (4)
N1	0.0461 (7)	0.0403 (6)	0.0486 (7)	0.0048 (5)	0.0165 (5)	0.0007 (5)
N2	0.0381 (6)	0.0334 (5)	0.0430 (6)	0.0003 (4)	0.0084 (5)	0.0007 (5)
N3	0.0346 (5)	0.0328 (5)	0.0449 (6)	0.0009 (4)	0.0057 (4)	0.0001 (5)
C1	0.0359 (6)	0.0413 (7)	0.0412 (7)	0.0013 (5)	0.0099 (5)	-0.0026 (5)
C2	0.0387 (7)	0.0566 (9)	0.0431 (7)	0.0007 (6)	0.0058 (6)	-0.0020 (7)
C3	0.0413 (8)	0.0607 (10)	0.0635 (10)	0.0099 (7)	0.0064 (7)	0.0075 (8)
C4	0.0522 (9)	0.0548 (10)	0.0701 (11)	0.0102 (7)	0.0230 (8)	-0.0050 (8)
C5	0.0601 (10)	0.0541 (9)	0.0475 (8)	-0.0007 (7)	0.0181 (7)	-0.0102 (7)
C6	0.0450 (7)	0.0467 (8)	0.0406 (7)	-0.0027 (6)	0.0068 (6)	-0.0020 (6)
C7	0.0633 (10)	0.0719 (12)	0.0477 (9)	0.0029 (9)	-0.0061 (7)	0.0019 (8)
C8	0.0576 (10)	0.0985 (16)	0.0443 (8)	0.0030 (10)	-0.0039 (7)	-0.0096 (9)
C9	0.0317 (6)	0.0387 (7)	0.0406 (6)	0.0035 (5)	0.0045 (5)	-0.0020 (6)
C10	0.0364 (6)	0.0353 (6)	0.0451 (7)	0.0020 (5)	0.0058 (5)	-0.0054 (5)
C11	0.0628 (10)	0.0439 (9)	0.0786 (11)	-0.0061 (7)	0.0292 (8)	-0.0192 (8)

Geometric parameters (Å, °)

Ni1—N2 ⁱ	1.8397 (11)	C3—H3	0.9300
Ni1—N2	1.8397 (11)	C4—C5	1.375 (2)
Ni1—N3 ⁱ	1.8779 (11)	C4—H4	0.9300
Ni1—N3	1.8779 (11)	C5—C6	1.384 (2)
O1—N2	1.3817 (15)	C5—H5	0.9300
O1—H1O	0.858 (9)	C6—C7	1.504 (2)
O2—N3	1.3181 (14)	C7—H7A	0.9600
N1—C9	1.3575 (18)	C7—H7B	0.9600
N1—C1	1.4389 (17)	C7—H7C	0.9600
N1—H1N	0.824 (14)	C8—H8A	0.9600
N2—C9	1.3028 (19)	C8—H8B	0.9600
N3—C10	1.3111 (17)	C8—H8C	0.9600
C1—C2	1.387 (2)	C9—C10	1.470 (2)
C1—C6	1.3979 (19)	C10—C11	1.486 (2)
C2—C3	1.392 (2)	C11—H11A	0.9600
C2—C8	1.513 (2)	C11—H11B	0.9600
C3—C4	1.374 (2)	C11—H11C	0.9600
N2 ⁱ —Ni1—N2	180.0	C4—C5—H5	119.2
N2 ⁱ —Ni1—N3 ⁱ	82.38 (5)	C6—C5—H5	119.2
N2—Ni1—N3 ⁱ	97.62 (5)	C5—C6—C1	117.53 (14)
N2 ⁱ —Ni1—N3	97.62 (5)	C5—C6—C7	121.19 (14)

N2—Ni1—N3	82.38 (5)	C1—C6—C7	121.26 (13)
N3 ⁱ —Ni1—N3	180.0	C6—C7—H7A	109.5
N2—O1—H1O	99.1 (15)	C6—C7—H7B	109.5
C9—N1—C1	124.51 (13)	H7A—C7—H7B	109.5
C9—N1—H1N	109.3 (12)	C6—C7—H7C	109.5
C1—N1—H1N	121.9 (12)	H7A—C7—H7C	109.5
C9—N2—O1	116.31 (12)	H7B—C7—H7C	109.5
C9—N2—Ni1	116.99 (11)	C2—C8—H8A	109.5
O1—N2—Ni1	126.55 (9)	C2—C8—H8B	109.5
C10—N3—O2	120.48 (11)	H8A—C8—H8B	109.5
C10—N3—Ni1	116.60 (9)	C2—C8—H8C	109.5
O2—N3—Ni1	122.85 (8)	H8A—C8—H8C	109.5
C2—C1—C6	122.03 (13)	H8B—C8—H8C	109.5
C2—C1—N1	120.00 (13)	N2—C9—N1	121.96 (14)
C6—C1—N1	117.88 (13)	N2—C9—C10	113.00 (12)
C1—C2—C3	118.10 (14)	N1—C9—C10	124.96 (13)
C1—C2—C8	122.03 (14)	N3—C10—C9	110.90 (12)
C3—C2—C8	119.87 (15)	N3—C10—C11	122.18 (13)
C4—C3—C2	120.84 (16)	C9—C10—C11	126.81 (12)
C4—C3—H3	119.6	C10—C11—H11A	109.5
C2—C3—H3	119.6	C10—C11—H11B	109.5
C3—C4—C5	119.93 (14)	H11A—C11—H11B	109.5
C3—C4—H4	120.0	C10—C11—H11C	109.5
C5—C4—H4	120.0	H11A—C11—H11C	109.5
C4—C5—C6	121.55 (15)	H11B—C11—H11C	109.5
N3 ⁱ —Ni1—N2—C9	-177.23 (10)	C4—C5—C6—C7	177.73 (16)
N3—Ni1—N2—C9	2.77 (10)	C2—C1—C6—C5	0.3 (2)
N3 ⁱ —Ni1—N2—O1	-1.92 (12)	N1—C1—C6—C5	-176.24 (14)
N3—Ni1—N2—O1	178.08 (12)	C2—C1—C6—C7	-178.00 (15)
N2 ⁱ —Ni1—N3—C10	179.36 (10)	N1—C1—C6—C7	5.4 (2)
N2—Ni1—N3—C10	-0.64 (10)	O1—N2—C9—N1	3.3 (2)
N2 ⁱ —Ni1—N3—O2	-3.65 (12)	Ni1—N2—C9—N1	179.15 (10)
N2—Ni1—N3—O2	176.35 (12)	O1—N2—C9—C10	-179.93 (11)
C9—N1—C1—C2	57.9 (2)	Ni1—N2—C9—C10	-4.13 (15)
C9—N1—C1—C6	-125.45 (16)	C1—N1—C9—N2	-150.05 (14)
C6—C1—C2—C3	0.6 (2)	C1—N1—C9—C10	33.6 (2)
N1—C1—C2—C3	177.13 (14)	O2—N3—C10—C9	-178.35 (11)
C6—C1—C2—C8	-178.83 (15)	Ni1—N3—C10—C9	-1.29 (15)
N1—C1—C2—C8	-2.3 (2)	O2—N3—C10—C11	-1.9 (2)
C1—C2—C3—C4	-1.4 (2)	Ni1—N3—C10—C11	175.16 (12)
C8—C2—C3—C4	178.11 (16)	N2—C9—C10—N3	3.42 (17)
C2—C3—C4—C5	1.1 (3)	N1—C9—C10—N3	-179.97 (13)
C3—C4—C5—C6	-0.1 (3)	N2—C9—C10—C11	-172.82 (14)
C4—C5—C6—C1	-0.6 (2)	N1—C9—C10—C11	3.8 (2)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

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>
O1—H1O \cdots O2 ⁱ	0.86 (1)	1.65 (1)	2.4972 (14)	171 (2)
N1—H1N \cdots O1	0.82 (1)	2.20 (2)	2.6361 (16)	113 (1)

Symmetry code: (i) $-x+1, -y+1, -z+1$.