Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

cis-(Acetato-κ^2O,O')(5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane-κ^4N,N',N'',N''')nickel(II) perchlorate monohydrate

Tapashi G. Roy,[a]‡ Debashis Palit,[a] Babul Chandra Nath,[a] Seik Weng Ng[b,c] and Edward R. T. Tiekink[b]*

[a]Department of Chemistry, University of Chittagong, Chittagong 4331, Bangladesh, [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia, and [c]Chemistry Department, Faculty of Science, King Abdulaziz University, PO Box 80203, Jeddah, Saudi Arabia
Correspondence e-mail: edward.tiekink@gmail.com

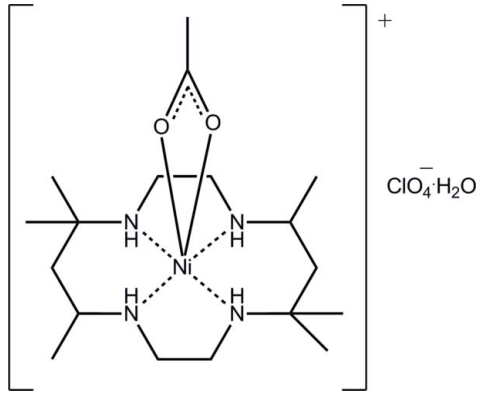
Received 26 March 2012; accepted 27 March 2012

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; disorder in solvent or counterion; R factor = 0.067; wR factor = 0.162; data-to-parameter ratio = 14.5.

The complete cation in the title hydrated molecular salt, $[Ni(CH_3CO_2)(C_{16}H_{36}N_4)]ClO_4\cdot H_2O$, is generated by the application of crystallographic twofold symmetry; the perchlorate anion and water molecule are each disordered around a twofold axis. The Ni^{II} atom exists within a *cis*-N_4O_2 donor set based on a strongly distorted octahedron and defined by the four N atoms of the macrocyclic ligand and two O atoms of a symmetrically coordinating acetate ligand. In the crystal, hydrogen bonding (water–acetate/perchlorate O—H···O and amine–perchlorate N—H···O) leads to layers in the *ab* plane. The layers stack along the *c* axis, being connected by C—H···O(water) interactions. The crystal studied was found to be a non-merohedral twin; the minor component refined to 15.9 (6)%.

Related literature

For background to macrocyclic complexes, see: Hazari *et al.* (2010). For a related structure, see: Roy *et al.* (2012). For the treatment of data from twinned crystals, see: Spek (2009).



Experimental

Crystal data

$[Ni(C_2H_3O_2)(C_{16}H_{36}N_4)]ClO_4\cdot H_2O$
M_r = 519.71
Monoclinic, $C2/c$
a = 9.4041 (2) Å
b = 15.9593 (4) Å
c = 16.0721 (6) Å
β = 96.534 (3)°
V = 2396.48 (12) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.58 mm^{-1}
T = 100 K
0.25 × 0.20 × 0.15 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.850, T_{max} = 1.000
5502 measured reflections
2480 independent reflections
2286 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.162
S = 1.09
2480 reflections
171 parameters
45 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—O1	2.118 (2)	Ni—N2	2.136 (3)
Ni—N1	2.089 (3)		
O1^i—Ni—O1	62.28 (13)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.88	2.47	3.289 (14)	154
N1—H1···O3^i	0.88	2.35	3.181 (9)	158
N2—H2···O4^{ii}	0.88	2.50	3.276 (8)	148
O1w—H1w1···O1^{iii}	0.84	1.94	2.754 (11)	163
O1w—H1w2···O2	0.84	2.14	2.950 (18)	163
C3—H3*A*···O1*W*^{iv}	0.99	2.14	3.057 (13)	153

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

‡ Additional correspondence author, e-mail: tapashir@yahoo.com.

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors are grateful to the University Grand Commission (UGC), Bangladesh, for a research fellowship to BCN, and to the Research Support and Publication Division, University of Chittagong, for a research grant (5320/res/pub-CU/2012) to TGR. We also thank the Ministry of Higher Education (Malaysia) for funding structural studies through the High-Impact Research scheme (UM.C/HIR/MOHE/SC/12).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6703).

References

Agilent (2011). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Hazari, S. K. S., Roy, T. G., Barua, K. K., Anwar, N., Zukerman-Schpector, J. & Tiekink, E. R. T. (2010). *Appl. Organomet. Chem.* **24**, 878–887.
Roy, T. G., Hazari, S. K. S., Nath, B. C., Ng, S. W. & Tiekink, E. R. T. (2012). *Acta Cryst.* E**68**, m494–m495.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2012). E**68**, m525–m526 [https://doi.org/10.1107/S1600536812013232]

cis-(Acetato-κ^2O,O')(5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane-κ^4N,N',N'',N''')nickel(II) perchlorate monohydrate

Tapashi G. Roy, Debashis Palit, Babul Chandra Nath, Seik Weng Ng and Edward R. T. Tiekink

S1. Comment

As a continuation of systematic studies into the synthesis, characterization and biological activities of substituted tetraazamacrocyclic ligands and their metal complexes (Hazari *et al.*, 2010; Roy *et al.*, 2012), crystals of the title hydrated salt, (I), were isolated and characterized crystallographically.

The asymmetric unit of (I) comprises half a NiL(O_2CMe) cation, Fig. 1, as this is has crystallographic twofold symmetry, half a perchlorate anion (this is disordered about a twofold axis) and half a water molecule of solvation (this is also disordered about a twofold axis); where *L* is 5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane. The Ni^{II} atom exists within a *cis*-N_4O_2 donor set defined by the four N atoms of the macrocyclic ligand and two acetate-O atoms, Table 1. The coordination geometry is based on an octahedron. There are significant distortions from the ideal geometry owing in part to the restricted bite angle of the acetate ligand as manifested in the O1—Ni—$O1^i$ angle of 62.28 (13)°; symmetry operation *i*: 1 - *x*, *y*, 3/2 - *z*. In particular, this bite angle restricts the putative *trans* O1—Ni—O1 angle to 158.84 (12)°; the N2—Ni—$N2^i$ angle = 175.92 (17)°.

In the crystal packing, the water molecule forms O—H···O hydrogen bonds to the acetate-O1 and perchlorate-O2 atoms, while the amine-H atoms form hydrogen bonds to perchlorate-O atoms; the N1—H atom is bifurcated, Table 2. The hydrogen bonding leads to layers in the *ab* plane, Fig. 2. Layers are connected along the *c* axis by C—H···O(water) interactions, Fig. 3 and Table 2.

S2. Experimental

The title complex, (I), was prepared by the anion exchange reaction of [NiL(O_2CMe)][O_2CMe] with perchlorate, where *L* is 5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane. Thus, [NiL(O_2CMe)][O_2CMe] (0.495 g, 1.0 mmol) was dissolved in hot methanol (40 ml) and sodium perchlorate hexahydrate (0.460 g, 2.0 mmol) added. The reaction mixture was heated for 15 min. During heating a blue product separated out. After cooling at room temperature for 30 min, the product, (I), was filtered off, washed with methanol followed by diethyl ether and dried in a desiccator over silica-gel. Light-purple prisms of (I) were obtained from slow evaporation of its methanol solution. Yield 65%. *M*.pt: 512–513 K. Anal. Calc for $C_{18}H_{41}ClN_4NiO_7$: C, 41.68; H, 7.97; N, 10.81; Ni, 11.18%. Found: C, 44.53; H, 7.72; N, 10.78; Ni, 11.01%. FT—IR (KBr, cm^{-1}): 1598 ν(O_2C), 3202 ν(N—H), 2981 ν(C—H), 1369 ν(CH_3), 1177 ν(C—C), 520 (Ni—N), 1126, 623 ν(ClO_4).

S3. Refinement

The H-atoms were placed in calculated positions (O—H = 0.84, N—H = 0.88 and C—H = 0.98–1.00 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) = 1.2–1.5U_{equiv}(carrier atom). The perchlorate and water molecules are disordered across a twofold axis. The Cl—O bonds lengths were restrained to 0.01 Å of each

other, as were the O···O contact distances. The anisotropic displacement parameters were restrained to be nearly isotropic. Finally, the methyl-H atoms of the acetate group are disordered over two positions of equal weight. The crystal studied is a non-merohedral twin. The twin domains were separated by the *TwinRotMat* routine in *PLATON* (Spek, 2009). The minor component refined to 15.9 (6)%.

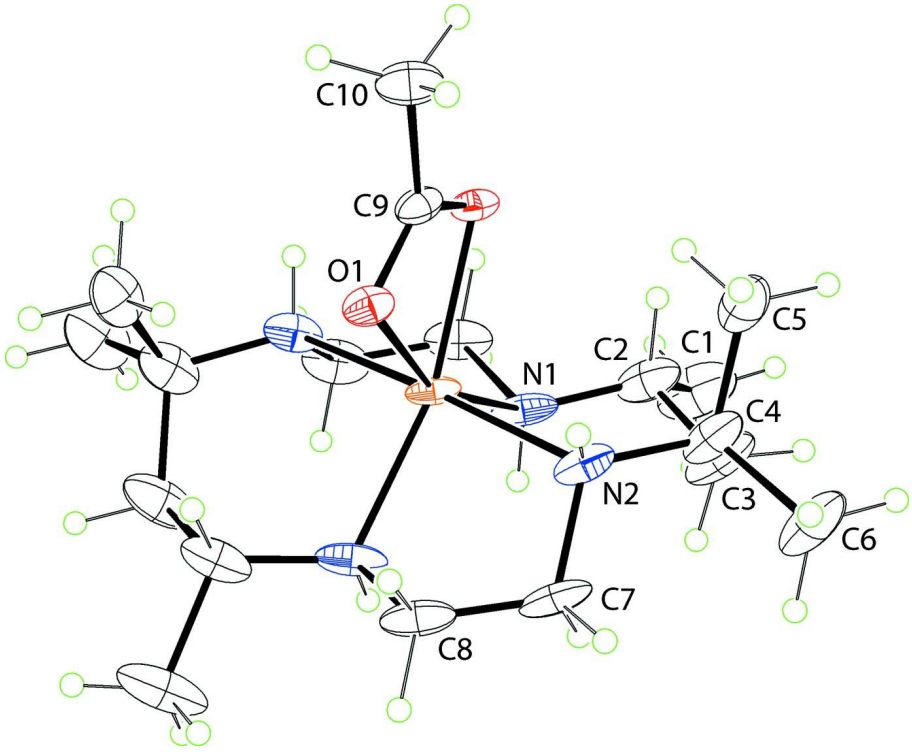


Figure 1

The molecular structure of the cation in (I) showing displacement ellipsoids at the 50% probability level. The cation has crystallographic twofold symmetry and unlabelled atoms are generated by the symmetry operation 1 - *x*, *y*, 3/2 - *z*.

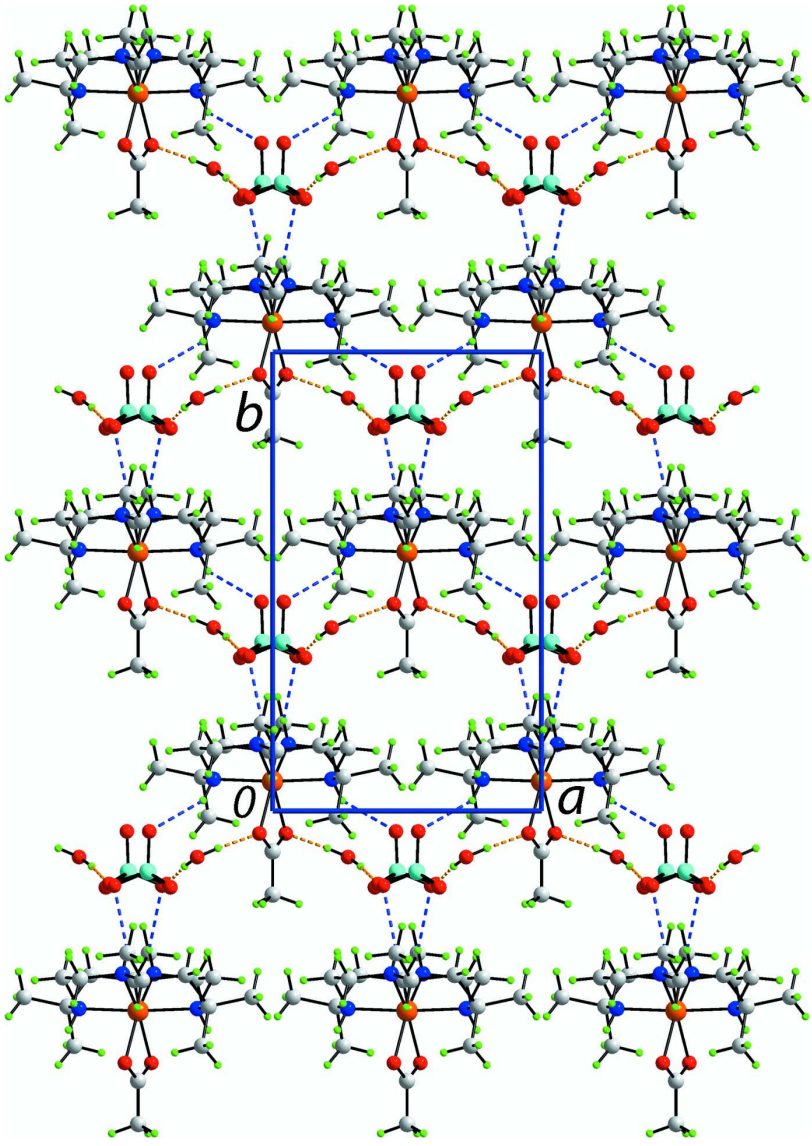


Figure 2

A view of the supramolecular layer in the *ab* plane in (I). The O—H···O and N—H···O hydrogen bonds are shown as orange and blue dashed lines, respectively. The illustrated perchlorate and water molecules are disordered about a twofold axis and each is present 50% of the time.

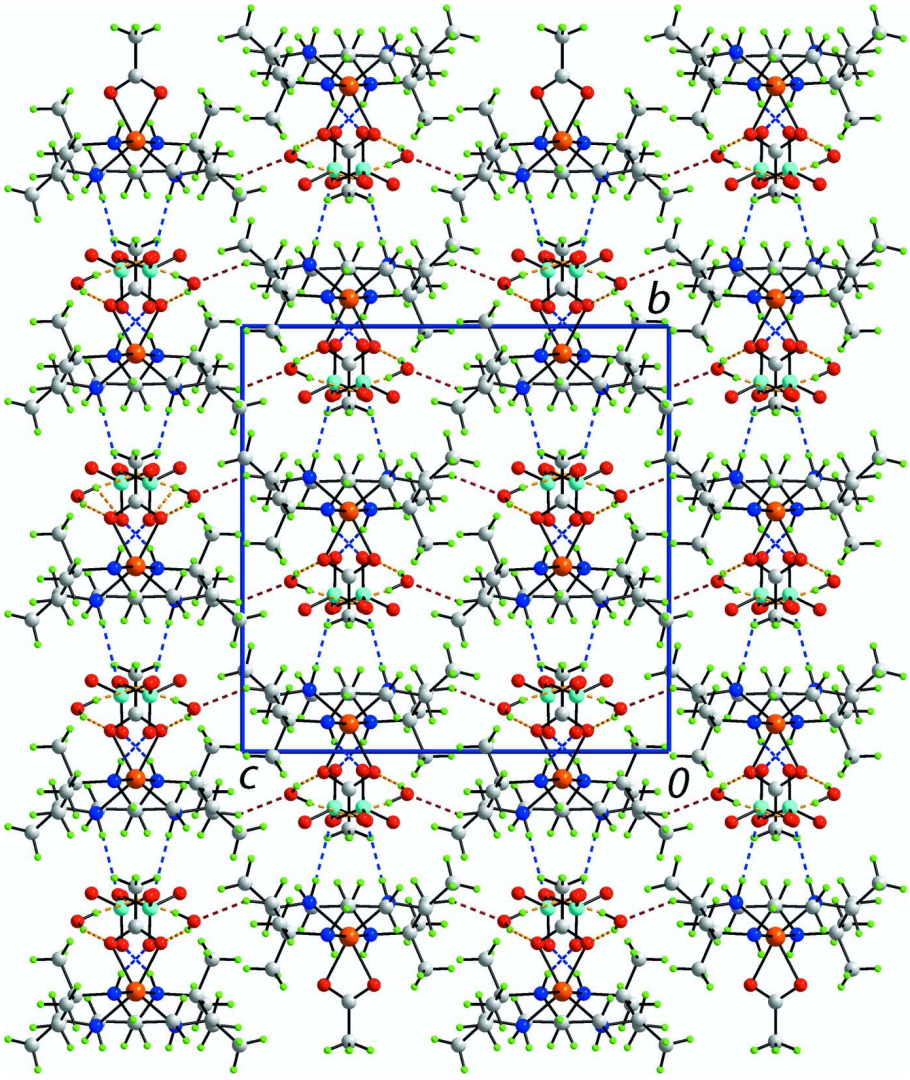


Figure 3

A view of the unit-cell contents in projection down the *a* axis in (I). The O—H···O, N—H···O and C—H···O interactions are shown as orange, blue and brown dashed lines, respectively. The illustrated perchlorate and water molecules are disordered about a twofold axis and each is present 50% of the time.

cis-(Acetato-κ^2O,O')(5,5,7,12,12,14-hexamethyl- 1,4,8,11-tetraazacyclotetradecane- κ^4N,N',N'',N''')nickel(II) perchlorate monohydrate

Crystal data

$[Ni(C_2H_3O_2)(C_{16}H_{36}N_4]ClO_4·H_2O$
M_r = 519.71
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 9.4041 (2) Å
b = 15.9593 (4) Å
c = 16.0721 (6) Å
β = 96.534 (3)°
V = 2396.48 (12) Å^3
Z = 4

F(000) = 1112
D_x = 1.440 Mg m^{-3}
Cu *K*α radiation, λ = 1.54184 Å
Cell parameters from 3218 reflections
θ = 5.5–76.4°
μ = 2.58 mm^{-1}
T = 100 K
Prism, light-purple
0.25 × 0.20 × 0.15 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.850, T_{max} = 1.000
5502 measured reflections
2480 independent reflections
2286 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 76.6°, θ_{min} = 5.5°
h = −11→11
k = −18→19
l = −3→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.162
S = 1.09
2480 reflections
171 parameters
45 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
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 9.9029P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Ni	0.5000	0.56446 (4)	0.7500	0.0300 (3)	
Cl1	0.4637 (3)	0.86782 (11)	0.71627 (15)	0.0559 (6)	0.50
O2	0.6083 (10)	0.8409 (9)	0.7174 (9)	0.197 (12)	0.50
O3	0.4105 (13)	0.8372 (5)	0.7910 (6)	0.111 (5)	0.50
O4	0.4585 (9)	0.9554 (3)	0.7181 (5)	0.085 (3)	0.50
O5	0.3800 (9)	0.8336 (6)	0.6466 (5)	0.111 (3)	0.50
O1W	0.7196 (13)	0.8995 (10)	0.8864 (7)	0.147 (6)	0.50
H1W1	0.7967	0.9187	0.8726	0.220*	0.50
H1W2	0.6729	0.8794	0.8433	0.220*	0.50
O1	0.4508 (2)	0.45087 (14)	0.80858 (16)	0.0318 (5)	
N1	0.5584 (4)	0.64503 (18)	0.6573 (2)	0.0459 (9)	
H1	0.5406	0.6967	0.6721	0.069*	
N2	0.2798 (3)	0.5692 (2)	0.6999 (2)	0.0412 (8)	
H2	0.2413	0.5227	0.7164	0.062*	
C1	0.5402 (7)	0.6902 (4)	0.5067 (4)	0.085 (2)	
H1A	0.6439	0.6833	0.5075	0.128*	

H1B	0.5188	0.7483	0.5206	0.128*	
H1C	0.4930	0.6766	0.4508	0.128*	
C2	0.4854 (5)	0.6312 (3)	0.5713 (3)	0.0546 (12)	
H2A	0.5042	0.5722	0.5546	0.066*	
C3	0.3231 (5)	0.6426 (3)	0.5688 (4)	0.0681 (16)	
H3A	0.2832	0.6489	0.5095	0.082*	
H3B	0.3059	0.6961	0.5973	0.082*	
C4	0.2374 (5)	0.5741 (3)	0.6076 (3)	0.0535 (12)	
C5	0.2620 (5)	0.4882 (3)	0.5708 (3)	0.0554 (11)	
H5A	0.3634	0.4733	0.5823	0.083*	
H5B	0.2345	0.4895	0.5102	0.083*	
H5C	0.2039	0.4465	0.5963	0.083*	
C6	0.0754 (5)	0.5945 (4)	0.5877 (4)	0.0749 (17)	
H6A	0.0558	0.6499	0.6102	0.112*	
H6B	0.0190	0.5521	0.6134	0.112*	
H6C	0.0493	0.5944	0.5269	0.112*	
C7	0.2205 (4)	0.6372 (3)	0.7477 (3)	0.0534 (12)	
H7A	0.1153	0.6310	0.7446	0.064*	
H7B	0.2415	0.6920	0.7229	0.064*	
C8	0.2843 (4)	0.6345 (3)	0.8372 (3)	0.0514 (11)	
H8A	0.2428	0.6799	0.8689	0.062*	
H8B	0.2614	0.5803	0.8624	0.062*	
C9	0.5000	0.4119 (3)	0.7500	0.0312 (10)	
C10	0.5000	0.3177 (3)	0.7500	0.0555 (16)	
H10A	0.4629	0.2973	0.8008	0.083*	0.50
H10B	0.5979	0.2973	0.7486	0.083*	0.50
H10C	0.4392	0.2973	0.7006	0.083*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni	0.0253 (4)	0.0131 (4)	0.0551 (6)	0.000	0.0195 (4)	0.000
Cl1	0.0651 (14)	0.0221 (8)	0.0840 (16)	0.0086 (8)	0.0232 (11)	−0.0024 (8)
O2	0.201 (15)	0.173 (14)	0.218 (15)	0.025 (9)	0.034 (10)	−0.029 (9)
O3	0.150 (9)	0.049 (5)	0.131 (8)	−0.031 (5)	0.013 (6)	0.048 (5)
O4	0.104 (7)	0.022 (3)	0.141 (8)	0.003 (3)	0.063 (5)	−0.004 (3)
O5	0.117 (7)	0.117 (7)	0.093 (6)	0.022 (6)	−0.005 (5)	−0.035 (5)
O1W	0.122 (9)	0.232 (15)	0.087 (7)	−0.090 (10)	0.017 (6)	−0.022 (8)
O1	0.0294 (12)	0.0186 (11)	0.0479 (14)	−0.0039 (9)	0.0068 (10)	0.0020 (9)
N1	0.0461 (19)	0.0204 (14)	0.079 (2)	0.0067 (13)	0.0404 (18)	0.0113 (14)
N2	0.0320 (15)	0.0318 (16)	0.063 (2)	0.0107 (13)	0.0194 (14)	0.0140 (14)
C1	0.090 (4)	0.070 (4)	0.106 (5)	0.023 (3)	0.058 (4)	0.052 (3)
C2	0.059 (3)	0.044 (2)	0.067 (3)	0.018 (2)	0.034 (2)	0.026 (2)
C3	0.061 (3)	0.065 (3)	0.083 (3)	0.034 (2)	0.029 (3)	0.044 (3)
C4	0.038 (2)	0.058 (3)	0.066 (3)	0.0198 (19)	0.0136 (19)	0.024 (2)
C5	0.041 (2)	0.070 (3)	0.054 (2)	0.014 (2)	0.0018 (19)	0.010 (2)
C6	0.044 (3)	0.094 (4)	0.087 (4)	0.032 (3)	0.010 (3)	0.031 (3)
C7	0.038 (2)	0.038 (2)	0.090 (3)	0.0211 (17)	0.033 (2)	0.014 (2)

C8	0.042 (2)	0.034 (2)	0.086 (3)	0.0081 (17)	0.042 (2)	0.002 (2)
C9	0.024 (2)	0.019 (2)	0.050 (3)	0.000	−0.001 (2)	0.000
C10	0.074 (4)	0.017 (2)	0.076 (4)	0.000	0.010 (3)	0.000

Geometric parameters (Å, º)

Ni—O1	2.118 (2)	C2—H2A	1.0000
Ni—N1^{i}	2.089 (3)	C3—C4	1.532 (7)
Ni—N1	2.089 (3)	C3—H3A	0.9900
Ni—O1^{i}	2.118 (2)	C3—H3B	0.9900
Ni—N2	2.136 (3)	C4—C5	1.521 (7)
Ni—N2^{i}	2.136 (3)	C4—C6	1.556 (6)
Cl1—O4	1.400 (5)	C5—H5A	0.9800
Cl1—O5	1.404 (6)	C5—H5B	0.9800
Cl1—O2	1.424 (8)	C5—H5C	0.9800
Cl1—O3	1.439 (7)	C6—H6A	0.9800
O1W—H1W1	0.8400	C6—H6B	0.9800
O1W—H1W2	0.8399	C6—H6C	0.9800
O1—C9	1.259 (3)	C7—C8	1.495 (7)
N1—C8^{i}	1.481 (5)	C7—H7A	0.9900
N1—C2	1.488 (6)	C7—H7B	0.9900
N1—H1	0.8800	C8—N1^{i}	1.481 (5)
N2—C7	1.475 (5)	C8—H8A	0.9900
N2—C4	1.493 (6)	C8—H8B	0.9900
N2—H2	0.8800	C9—O1^{i}	1.259 (3)
C1—C2	1.534 (6)	C9—C10	1.503 (7)
C1—H1A	0.9800	C10—H10A	0.9800
C1—H1B	0.9800	C10—H10B	0.9800
C1—H1C	0.9800	C10—H10C	0.9800
C2—C3	1.532 (6)		

N1^{i}—Ni—N1	104.01 (18)	C3—C2—H2A	108.2
N1^{i}—Ni—O1^{i}	158.84 (12)	C1—C2—H2A	108.2
N1—Ni—O1^{i}	96.96 (11)	C2—C3—C4	118.2 (3)
N1^{i}—Ni—O1	96.96 (11)	C2—C3—H3A	107.8
N1—Ni—O1	158.84 (12)	C4—C3—H3A	107.8
O1^{i}—Ni—O1	62.28 (13)	C2—C3—H3B	107.8
N1^{i}—Ni—N2	85.68 (14)	C4—C3—H3B	107.8
N1—Ni—N2	91.80 (13)	H3A—C3—H3B	107.1
O1^{i}—Ni—N2	96.56 (11)	N2—C4—C5	107.7 (3)
O1—Ni—N2	86.94 (11)	N2—C4—C3	110.4 (4)
N1^{i}—Ni—N2^{i}	91.80 (13)	C5—C4—C3	112.0 (4)
N1—Ni—N2^{i}	85.68 (14)	N2—C4—C6	111.1 (4)
O1^{i}—Ni—N2^{i}	86.94 (11)	C5—C4—C6	107.3 (5)
O1—Ni—N2^{i}	96.56 (11)	C3—C4—C6	108.4 (4)
N2—Ni—N2^{i}	175.92 (17)	C4—C5—H5A	109.5
O4—Cl1—O5	112.8 (5)	C4—C5—H5B	109.5
O4—Cl1—O2	109.7 (5)	H5A—C5—H5B	109.5

O5—Cl1—O2	109.9 (5)	C4—C5—H5C	109.5
O4—Cl1—O3	107.8 (4)	H5A—C5—H5C	109.5
O5—Cl1—O3	108.5 (5)	H5B—C5—H5C	109.5
O2—Cl1—O3	108.0 (5)	C4—C6—H6A	109.5
H1W1—O1W—H1W2	107.9	C4—C6—H6B	109.5
C9—O1—Ni	88.4 (2)	H6A—C6—H6B	109.5
C8^{i}—N1—C2	113.0 (3)	C4—C6—H6C	109.5
C8^{i}—N1—Ni	103.3 (3)	H6A—C6—H6C	109.5
C2—N1—Ni	116.1 (2)	H6B—C6—H6C	109.5
C8^{i}—N1—H1	108.0	N2—C7—C8	110.3 (3)
C2—N1—H1	108.0	N2—C7—H7A	109.6
Ni—N1—H1	108.0	C8—C7—H7A	109.6
C7—N2—C4	113.9 (3)	N2—C7—H7B	109.6
C7—N2—Ni	103.7 (3)	C8—C7—H7B	109.6
C4—N2—Ni	120.9 (2)	H7A—C7—H7B	108.1
C7—N2—H2	105.7	N1^{i}—C8—C7	110.0 (3)
C4—N2—H2	105.7	N1^{i}—C8—H8A	109.7
Ni—N2—H2	105.7	C7—C8—H8A	109.7
C2—C1—H1A	109.5	N1^{i}—C8—H8B	109.7
C2—C1—H1B	109.5	C7—C8—H8B	109.7
H1A—C1—H1B	109.5	H8A—C8—H8B	108.2
C2—C1—H1C	109.5	O1^{i}—C9—O1	120.9 (4)
H1A—C1—H1C	109.5	O1^{i}—C9—C10	119.6 (2)
H1B—C1—H1C	109.5	O1—C9—C10	119.6 (2)
N1—C2—C3	111.1 (4)	C9—C10—H10A	109.5
N1—C2—C1	112.4 (5)	C9—C10—H10B	109.5
C3—C2—C1	108.6 (4)	C9—C10—H10C	109.5
N1—C2—H2A	108.2		

N1^{i}—Ni—O1—C9	−175.68 (14)	O1—Ni—N2—C4	−123.0 (3)
N1—Ni—O1—C9	11.9 (4)	C8^{i}—N1—C2—C3	180.0 (3)
O1^{i}—Ni—O1—C9	0.0	Ni—N1—C2—C3	60.9 (4)
N2—Ni—O1—C9	99.05 (14)	C8^{i}—N1—C2—C1	−58.0 (4)
N2^{i}—Ni—O1—C9	−83.05 (14)	Ni—N1—C2—C1	−177.1 (3)
N1^{i}—Ni—N1—C8^{i}	109.4 (3)	N1—C2—C3—C4	−73.5 (6)
O1^{i}—Ni—N1—C8^{i}	−67.8 (3)	C1—C2—C3—C4	162.3 (5)
O1—Ni—N1—C8^{i}	−78.4 (5)	C7—N2—C4—C5	−161.4 (3)
N2—Ni—N1—C8^{i}	−164.6 (3)	Ni—N2—C4—C5	74.0 (4)
N2^{i}—Ni—N1—C8^{i}	18.6 (3)	C7—N2—C4—C3	76.1 (4)
N1^{i}—Ni—N1—C2	−126.3 (3)	Ni—N2—C4—C3	−48.5 (4)
O1^{i}—Ni—N1—C2	56.5 (3)	C7—N2—C4—C6	−44.2 (5)
O1—Ni—N1—C2	45.9 (5)	Ni—N2—C4—C6	−168.8 (3)
N2—Ni—N1—C2	−40.3 (3)	C2—C3—C4—N2	65.0 (6)
N2^{i}—Ni—N1—C2	142.9 (3)	C2—C3—C4—C5	−55.0 (7)
N1^{i}—Ni—N2—C7	10.5 (2)	C2—C3—C4—C6	−173.1 (5)
N1—Ni—N2—C7	−93.4 (2)	C4—N2—C7—C8	−172.0 (3)
O1^{i}—Ni—N2—C7	169.4 (2)	Ni—N2—C7—C8	−38.6 (4)
O1—Ni—N2—C7	107.7 (2)	N2—C7—C8—N1^{i}	60.1 (4)

N1^{i}—Ni—N2—C4	139.7 (3)	Ni—O1—C9—O1^{i}	0.0
N1—Ni—N2—C4	35.8 (3)	Ni—O1—C9—C10	180.000 (1)
O1^{i}—Ni—N2—C4	−61.4 (3)		

Symmetry code: (i) −*x*+1, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.88	2.47	3.289 (14)	154
N1—H1···O3^{i}	0.88	2.35	3.181 (9)	158
N2—H2···O4^{ii}	0.88	2.50	3.276 (8)	148
O1w—H1w1···O1^{iii}	0.84	1.94	2.754 (11)	163
O1w—H1w2···O2	0.84	2.14	2.950 (18)	163
C3—H3*A*···O1*W*^{iv}	0.99	2.14	3.057 (13)	153

Symmetry codes: (i) −*x*+1, *y*, −*z*+3/2; (ii) −*x*+1/2, *y*−1/2, −*z*+3/2; (iii) *x*+1/2, *y*+1/2, *z*; (iv) *x*−1/2, −*y*+3/2, *z*−1/2.