IUCrData

ISSN 2414-3146

Bis(2,2′:6′,2″-terpyridine-κ^3N,N',N'')nickel(II) bis(perchlorate) hemihydrate

Carolin Anderer,* Christian Näther and Wolfgang Bensch

Institut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Max-Eyth-Strasse 2, 24118 Kiel, Germany. *Correspondence e-mail: canderer@ac.uni-kiel.de

Received 3 June 2016
Accepted 21 June 2016

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

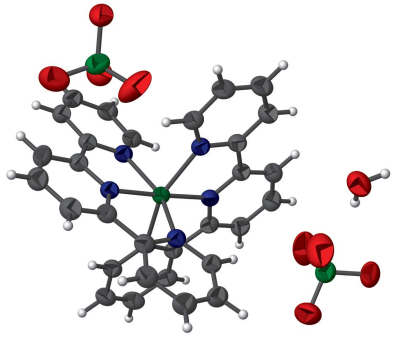
Keywords: crystal structure; nickel complex; octahedral coordination; hydrogen bonding.

CCDC reference: 1486868

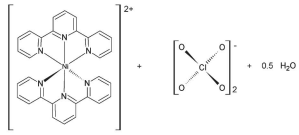
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $[Ni(C_{15}H_{11}N_3)_2](ClO_4)_2\cdot 0.5H_2O$, the Ni^{2+} cation is coordinated by two terpyridine ligands to form a discrete complex and the coordination polyhedron can be described as a slightly distorted octahedron. It crystallizes as a hemihydrate with two perchlorate anions to compensate the charges. In the crystal, one of the two crystallographically independent perchlorate anions is involved in O—H···O hydrogen bonding to the water molecules, where two inversion-related water molecules link two inversion-related perchlorate anions into a ring with an $R_4^2(12)$ loop. The O-atom position of the water molecule is only half occupied, *i.e.* only half of the anions are involved in hydrogen bonding. A similar arrangement of two anions is also observed for the second crystallographically independent perchlorate anion but no water molecules are located between the anions. The cationic complex and the perchlorate anions are additionally linked by a number of weak C—H···O hydrogen bonds, forming a three-dimensional supramolecular structure. The crystal structure of the monohydrate of the same complex has been reported [Baker *et al.* (1995). *Aust. J. Chem.* **48**, 1373–1378].

3D view



Chemical scheme



Structure description

Crystals of the title compound were obtained by the reaction of nickel perchlorate, terpyridine (terpy) and sodium trithioantimonate in H_2O during the synthesis of new thioantimonates containing Ni^{2+} cations. The title complex, Fig. 1, consists of an Ni^{2+} cation coordinated by two terpyridine ligands, two perchlorate anions and half a water molecule, all of them located in general positions. The Ni^{2+} coordination sphere can be described as an $NiNN_6$ slightly distorted octahedron (Fig. 1).

In the crystal structure, one of the two crystallographically independent perchlorate anions is involved in O—H···O hydrogen bonding to the water molecules, where two

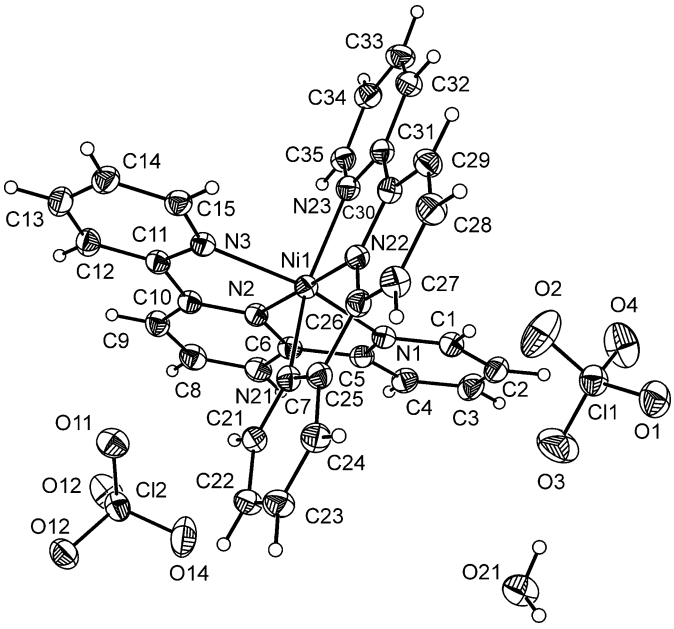


Figure 1
Molecular structure of the title compound, with atom labelling and displacement ellipsoids drawn at the 30% probability level.

water molecules link two perchlorate anions into a ring (Figs. 2 and 3, and Table 1). The shortest intermolecular O···O distances between the two anions within the ring is 5.273 (4) Å. It is noted that the oxygen position of the water molecule is only half occupied, *i.e.* only half of the anions are involved in hydrogen bonding. A similar arrangement of two anions is also observed for the second crystallographically independent perchlorate anion but no water molecules are located between the anions leading to a shorter intermolecular distance (O···O distance *ca* 4.82 Å; see Fig. 3). The cationic complex and the perchlorate anions are additionally linked by a number of weak C—H···O hydrogen bonds (Table 1), that lead to the formation of a three-dimensional supramolecular structure.

The crystal structure of bis(2,2′-terpyridine)nickel(II) diperchlorate monohydrate in space group $P2_1$ (compared to $P2_1/n$ for the title complex) has been reported by Baker *et al.* (1995). The structure of the nickel nitrate complex of terpy (Calatayud *et al.*, 2005) and the nickel pentathionate complex of terpy (Freire *et al.*, 2001), have also been reported. McMurtrie & Dance (2010) have reported the structure of a nickel sulfate complex of terpy.

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

D—H···A	D—H	H···A	D···A	D—H···A
O21—H21A···O3	0.84	2.01	2.772 (5)	151
O21—H21B···O1^{i}	0.84	2.08	2.845 (5)	150
C1—H1···O2	0.95	2.58	3.490 (4)	162
C2—H2···O4	0.95	2.59	3.292 (4)	131
C4—H4···O12^{ii}	0.95	2.32	3.127 (3)	142
C9—H9···O13^{iii}	0.95	2.53	3.417 (3)	155
C12—H12···O13^{iii}	0.95	2.66	3.539 (3)	155
C15—H15···O3^{iv}	0.95	2.45	3.265 (4)	144
C15—H15···O21^{iv}	0.95	2.48	3.124 (5)	125
C21—H21···O11	0.95	2.52	3.255 (3)	134
C21—H21···O14	0.95	2.60	3.319 (4)	133
C24—H24···O2^{v}	0.95	2.65	3.575 (4)	166
C32—H32···O1^{vi}	0.95	2.39	3.243 (3)	149
C34—H34···O11^{vii}	0.95	2.52	3.095 (3)	119

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

Synthesis and crystallization

Na_3SbS_3 was prepared by a reported procedure (Pompe & Pfitzner, 2013). $Ni(ClO_4)_2{\cdot}6H_2O$ (36.6 mg, 0.1 mmol), terpyridine (46.7 mg, 0.2 mmol) and Na_3SbS_3 (172.2 mg, 0.6 mmol) were reacted under solvothermal conditions in 2 ml H_2O at 443 K for 26.5 h in an 11 ml glass tube. After cooling to room temperature, the solid was filtered off, washed with water and ethanol and dried over silica gel. The product consists of red block-like crystals and a grey powder of unknown identity.

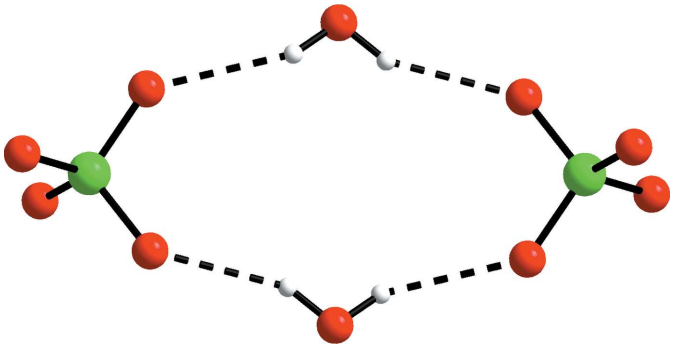


Figure 2
A view of the hydrogen-bonded $R_4^2(12)$ loop involving the water molecule and a perchlorate anion. Hydrogen bonds are shown as dashed lines (see Table 1).

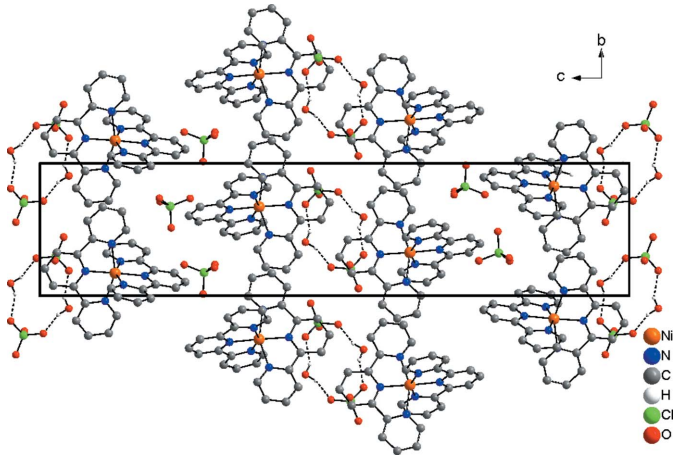


Figure 3
Crystal packing of the title compound, viewed along the *a* axis. Only the O—H···O hydrogen bonds are shown (dashed lines; see Table 1), and the C-bound H atoms have been omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Ni(C_{15}H_{11}N_3)_2](ClO_4)_2{\cdot}0.5H_2O$
M_r	733.15
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	150
a, b, c (Å)	8.7733 (2), 8.8342 (2), 39.4158 (10)
β (°)	94.150 (2)
V (Å^3)	3046.92 (12)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.88
Crystal size (mm)	0.1 × 0.08 × 0.07
Data collection	
Diffractometer	Stoe IPDS2
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	31108, 5107, 4629
R_{int}	0.047
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.585
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.107, 1.05
No. of reflections	5107
No. of parameters	436
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.41, −0.35

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *XP* in *SHELXTL* (Sheldrick, 2008), *DIAMOND* (Brandenburg, 1999) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The water position is not fully occupied, initially the occupancy factor was refined to be close to 0.5, and in the final cycles of refinement it was fixed at this value.

Acknowledgements

This work was supported by the State of Schleswig–Holstein.

References

Baker, A. T., Craig, D. C. & Rae, A. D. (1995). *Aust. J. Chem.* **48**, 1373–1378.
Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Calatayud, M. L., Sletten, J., Julve, M. & Castro, I. (2005). *J. Mol. Struct.* **741**, 121–128.
Freire, E., Baggio, S. & Baggio, R. (2001). *Aust. J. Chem.* **54**, 131–134.
McMurtrie, J. & Dance, I. (2010). *CrystEngComm*, **12**, 2700–2710.
Pompe, C. & Pfitzner, A. (2013). *Z. Anorg. Allg. Chem.* **639**, 296–300.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.
Stoe & Cie (2008). *X-AREA* and *X-RED32*. Stoe & Cie GmbH, Darmstadt, Germany.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2016). **1**, x161009 [https://doi.org/10.1107/S2414314616010099]

Bis(2,2′:6′,2″-terpyridine-κ^3N,N',N'')nickel(II) bis(perchlorate) hemihydrate

Carolin Anderer, Christian Näther and Wolfgang Bensch

Bis(2,2′:6′,2″-terpyridine-κ^3N,N',N'')nickel(II) bis(perchlorate) hemihydrate

Crystal data

$[Ni(C_{15}H_{11}N_3)_2](ClO_4)_2·0.5H_2O$
M_r = 733.15
Monoclinic, $P2_1/n$
a = 8.7733 (2) Å
b = 8.8342 (2) Å
c = 39.4158 (10) Å
β = 94.150 (2)°
V = 3046.92 (12) Å^3
Z = 4
$F(000)$ = 1500
D_x = 1.598 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 31108 reflections
θ = 1.0–24.6°
μ = 0.88 mm^{-1}
T = 150 K
Block, red
0.1 × 0.08 × 0.07 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
31108 measured reflections
5107 independent reflections
4629 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 24.6°, θ_{min} = 1.0°
h = −10→10
k = −10→10
l = −46→46

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 1.05
5107 reflections
436 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 1.5903P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Extinction correction: SHELXL2013 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0049 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ni1	0.64470 (3)	0.32729 (3)	0.37211 (2)	0.03402 (14)	
N1	0.4214 (2)	0.2406 (2)	0.36311 (5)	0.0367 (5)	
N2	0.6094 (2)	0.3488 (2)	0.32166 (5)	0.0366 (5)	
N3	0.8557 (2)	0.4161 (2)	0.35825 (5)	0.0377 (5)	
C1	0.3333 (3)	0.1807 (3)	0.38621 (7)	0.0405 (6)	
H1	0.3671	0.1865	0.4096	0.049*	
C2	0.1955 (3)	0.1112 (3)	0.37714 (7)	0.0462 (6)	
H2	0.1355	0.0697	0.3940	0.055*	
C3	0.1470 (3)	0.1030 (3)	0.34324 (8)	0.0510 (7)	
H3	0.0529	0.0550	0.3364	0.061*	
C4	0.2352 (3)	0.1647 (3)	0.31918 (7)	0.0457 (6)	
H4	0.2030	0.1594	0.2957	0.055*	
C5	0.3716 (3)	0.2343 (3)	0.32991 (6)	0.0387 (5)	
C6	0.4745 (3)	0.3055 (3)	0.30633 (6)	0.0387 (5)	
C7	0.4415 (3)	0.3280 (3)	0.27179 (7)	0.0460 (6)	
H7	0.3457	0.2986	0.2611	0.055*	
C8	0.5519 (3)	0.3946 (3)	0.25334 (7)	0.0492 (6)	
H8	0.5320	0.4107	0.2296	0.059*	
C9	0.6910 (3)	0.4380 (3)	0.26923 (7)	0.0455 (6)	
H9	0.7670	0.4837	0.2567	0.055*	
C10	0.7167 (3)	0.4133 (3)	0.30368 (6)	0.0385 (5)	
C11	0.8586 (3)	0.4522 (3)	0.32483 (6)	0.0388 (5)	
C12	0.9847 (3)	0.5194 (3)	0.31204 (7)	0.0447 (6)	
H12	0.9841	0.5449	0.2886	0.054*	
C13	1.1117 (3)	0.5490 (3)	0.33388 (8)	0.0499 (7)	
H13	1.1991	0.5962	0.3257	0.060*	
C14	1.1105 (3)	0.5093 (3)	0.36773 (7)	0.0466 (6)	
H14	1.1972	0.5274	0.3830	0.056*	
C15	0.9805 (3)	0.4427 (3)	0.37890 (7)	0.0410 (6)	
H15	0.9799	0.4148	0.4022	0.049*	
N21	0.5653 (2)	0.5392 (2)	0.38896 (5)	0.0373 (5)	
N22	0.6842 (2)	0.3128 (2)	0.42262 (5)	0.0355 (4)	
N23	0.7339 (2)	0.1055 (2)	0.37814 (5)	0.0376 (5)	
C21	0.5004 (3)	0.6500 (3)	0.36951 (7)	0.0401 (6)	
H21	0.4927	0.6377	0.3455	0.048*	
C22	0.4445 (3)	0.7810 (3)	0.38307 (7)	0.0461 (6)	
H22	0.3987	0.8569	0.3686	0.055*	
C23	0.4558 (3)	0.8001 (3)	0.41779 (8)	0.0497 (7)	
H23	0.4172	0.8891	0.4276	0.060*	
C24	0.5246 (3)	0.6873 (3)	0.43832 (7)	0.0457 (6)	
H24	0.5351	0.6990	0.4623	0.055*	
C25	0.5774 (3)	0.5578 (3)	0.42313 (6)	0.0386 (5)	
C26	0.6493 (3)	0.4290 (3)	0.44239 (6)	0.0385 (5)	
C27	0.6803 (3)	0.4232 (3)	0.47740 (7)	0.0457 (6)	
H27	0.6565	0.5064	0.4914	0.055*	

C28	0.7466 (3)	0.2937 (3)	0.49150 (7)	0.0489 (6)	
H28	0.7677	0.2870	0.5154	0.059*	
C29	0.7823 (3)	0.1735 (3)	0.47092 (7)	0.0448 (6)	
H29	0.8280	0.0842	0.4804	0.054*	
C30	0.7498 (3)	0.1868 (3)	0.43622 (7)	0.0385 (6)	
C31	0.7804 (3)	0.0699 (3)	0.41065 (6)	0.0378 (5)	
C32	0.8504 (3)	−0.0664 (3)	0.41903 (7)	0.0440 (6)	
H32	0.8837	−0.0885	0.4420	0.053*	
C33	0.8715 (3)	−0.1704 (3)	0.39353 (8)	0.0493 (7)	
H33	0.9208	−0.2642	0.3987	0.059*	
C34	0.8201 (3)	−0.1362 (3)	0.36048 (7)	0.0460 (6)	
H34	0.8312	−0.2070	0.3427	0.055*	
C35	0.7523 (3)	0.0024 (3)	0.35372 (7)	0.0403 (6)	
H35	0.7172	0.0259	0.3310	0.048*	
Cl1	0.21251 (8)	0.20342 (8)	0.47263 (2)	0.04847 (19)	
O1	0.1600 (3)	0.2159 (3)	0.50604 (5)	0.0631 (6)	
O2	0.3705 (3)	0.2339 (4)	0.47414 (7)	0.0918 (9)	
O3	0.1348 (4)	0.3097 (3)	0.45068 (7)	0.0924 (9)	
O4	0.1820 (3)	0.0561 (3)	0.45948 (6)	0.0825 (8)	
Cl2	0.48296 (8)	0.82419 (8)	0.27874 (2)	0.0496 (2)	
O11	0.6161 (2)	0.7635 (3)	0.29712 (5)	0.0585 (5)	
O12	0.4709 (3)	0.7648 (3)	0.24492 (6)	0.0734 (7)	
O13	0.4980 (3)	0.9858 (2)	0.27707 (6)	0.0628 (6)	
O14	0.3498 (3)	0.7874 (4)	0.29620 (7)	0.0866 (9)	
O21	0.0023 (5)	0.5946 (5)	0.45066 (10)	0.0582 (10)	0.5
H21A	0.0460	0.5156	0.4582	0.049 (17)*	0.5
H21B	−0.0185	0.6406	0.4684	0.07 (2)*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0341 (2)	0.0325 (2)	0.0354 (2)	−0.00023 (12)	0.00246 (13)	0.00065 (12)
N1	0.0369 (10)	0.0337 (11)	0.0396 (11)	0.0023 (9)	0.0036 (8)	0.0005 (9)
N2	0.0376 (11)	0.0320 (10)	0.0407 (11)	0.0006 (8)	0.0053 (9)	−0.0008 (8)
N3	0.0371 (10)	0.0336 (11)	0.0427 (11)	0.0018 (9)	0.0045 (9)	−0.0002 (9)
C1	0.0405 (13)	0.0373 (13)	0.0439 (14)	0.0008 (10)	0.0058 (11)	0.0009 (11)
C2	0.0388 (13)	0.0462 (15)	0.0546 (16)	−0.0018 (12)	0.0104 (11)	−0.0004 (13)
C3	0.0361 (13)	0.0532 (17)	0.0635 (18)	−0.0058 (12)	0.0033 (12)	−0.0048 (14)
C4	0.0400 (14)	0.0490 (16)	0.0474 (15)	−0.0003 (11)	−0.0013 (11)	−0.0031 (12)
C5	0.0357 (12)	0.0365 (13)	0.0436 (14)	0.0025 (10)	0.0011 (10)	−0.0006 (11)
C6	0.0387 (13)	0.0357 (13)	0.0414 (14)	0.0024 (10)	0.0011 (10)	−0.0016 (10)
C7	0.0491 (15)	0.0470 (16)	0.0407 (14)	0.0033 (12)	−0.0043 (12)	0.0016 (11)
C8	0.0591 (17)	0.0502 (16)	0.0382 (14)	0.0047 (13)	0.0017 (12)	0.0033 (12)
C9	0.0516 (15)	0.0457 (15)	0.0402 (14)	0.0019 (12)	0.0102 (11)	0.0045 (11)
C10	0.0400 (13)	0.0335 (12)	0.0425 (13)	0.0024 (10)	0.0061 (10)	−0.0005 (10)
C11	0.0420 (13)	0.0311 (12)	0.0440 (14)	0.0024 (10)	0.0076 (10)	−0.0005 (10)
C12	0.0467 (14)	0.0404 (14)	0.0484 (14)	−0.0030 (11)	0.0130 (11)	−0.0022 (12)
C13	0.0436 (14)	0.0426 (15)	0.0653 (18)	−0.0052 (12)	0.0163 (13)	−0.0034 (13)

C14	0.0374 (13)	0.0431 (15)	0.0594 (17)	−0.0023 (11)	0.0036 (11)	−0.0054 (12)
C15	0.0388 (13)	0.0373 (14)	0.0468 (14)	0.0017 (11)	0.0021 (11)	−0.0023 (11)
N21	0.0366 (10)	0.0354 (11)	0.0401 (11)	−0.0023 (8)	0.0045 (8)	0.0009 (9)
N22	0.0341 (10)	0.0323 (10)	0.0403 (11)	−0.0006 (8)	0.0038 (8)	0.0001 (8)
N23	0.0353 (10)	0.0361 (11)	0.0414 (11)	−0.0021 (9)	0.0023 (8)	−0.0002 (9)
C21	0.0385 (13)	0.0379 (13)	0.0439 (14)	−0.0006 (10)	0.0024 (11)	0.0051 (11)
C22	0.0441 (14)	0.0358 (14)	0.0582 (17)	0.0043 (11)	0.0019 (12)	0.0046 (12)
C23	0.0521 (15)	0.0382 (14)	0.0590 (17)	0.0051 (12)	0.0063 (13)	−0.0046 (12)
C24	0.0475 (15)	0.0430 (15)	0.0469 (15)	0.0000 (12)	0.0060 (12)	−0.0038 (12)
C25	0.0378 (12)	0.0356 (13)	0.0426 (13)	−0.0017 (10)	0.0050 (10)	−0.0009 (10)
C26	0.0363 (12)	0.0379 (13)	0.0416 (13)	−0.0036 (10)	0.0046 (10)	−0.0007 (11)
C27	0.0501 (15)	0.0466 (15)	0.0403 (14)	−0.0015 (12)	0.0035 (11)	−0.0035 (11)
C28	0.0516 (15)	0.0566 (17)	0.0380 (14)	0.0006 (13)	0.0007 (12)	0.0038 (12)
C29	0.0491 (15)	0.0432 (15)	0.0418 (14)	0.0030 (11)	0.0020 (12)	0.0066 (11)
C30	0.0353 (12)	0.0374 (13)	0.0428 (14)	−0.0029 (10)	0.0032 (10)	0.0025 (10)
C31	0.0348 (12)	0.0344 (13)	0.0442 (14)	−0.0024 (10)	0.0032 (10)	0.0003 (10)
C32	0.0412 (13)	0.0394 (14)	0.0510 (15)	0.0009 (11)	−0.0004 (11)	0.0062 (12)
C33	0.0459 (15)	0.0358 (14)	0.0654 (18)	0.0050 (11)	−0.0009 (13)	0.0005 (12)
C34	0.0420 (14)	0.0385 (14)	0.0573 (16)	0.0003 (11)	0.0038 (12)	−0.0079 (12)
C35	0.0375 (12)	0.0382 (14)	0.0450 (14)	−0.0021 (10)	0.0023 (10)	−0.0038 (11)
Cl1	0.0498 (4)	0.0530 (4)	0.0425 (4)	−0.0029 (3)	0.0024 (3)	0.0012 (3)
O1	0.0762 (14)	0.0714 (14)	0.0434 (11)	0.0017 (12)	0.0152 (10)	0.0032 (10)
O2	0.0523 (13)	0.148 (3)	0.0765 (17)	−0.0239 (16)	0.0113 (12)	−0.0276 (18)
O3	0.121 (2)	0.095 (2)	0.0606 (15)	0.0389 (17)	0.0048 (15)	0.0256 (14)
O4	0.123 (2)	0.0598 (15)	0.0651 (14)	−0.0260 (14)	0.0079 (14)	−0.0111 (12)
Cl2	0.0536 (4)	0.0471 (4)	0.0472 (4)	−0.0079 (3)	−0.0026 (3)	0.0082 (3)
O11	0.0604 (12)	0.0606 (13)	0.0524 (12)	0.0031 (10)	−0.0095 (9)	0.0025 (10)
O12	0.1052 (18)	0.0608 (14)	0.0502 (12)	−0.0133 (13)	−0.0219 (12)	−0.0002 (11)
O13	0.0817 (15)	0.0422 (11)	0.0671 (13)	0.0034 (10)	0.0240 (11)	0.0037 (10)
O14	0.0584 (14)	0.115 (2)	0.0859 (18)	−0.0224 (14)	0.0042 (12)	0.0435 (16)
O21	0.072 (3)	0.054 (3)	0.048 (2)	0.009 (2)	−0.002 (2)	−0.005 (2)

Geometric parameters (Å, º)

Ni1—N2	1.999 (2)	N22—C26	1.338 (3)
Ni1—N22	2.000 (2)	N22—C30	1.347 (3)
Ni1—N1	2.110 (2)	N23—C35	1.343 (3)
Ni1—N23	2.117 (2)	N23—C31	1.353 (3)
Ni1—N3	2.119 (2)	C21—C22	1.379 (4)
Ni1—N21	2.120 (2)	C21—H21	0.9500
N1—C1	1.345 (3)	C22—C23	1.375 (4)
N1—C5	1.350 (3)	C22—H22	0.9500
N2—C6	1.345 (3)	C23—C24	1.393 (4)
N2—C10	1.345 (3)	C23—H23	0.9500
N3—C15	1.337 (3)	C24—C25	1.387 (4)
N3—C11	1.357 (3)	C24—H24	0.9500
C1—C2	1.379 (4)	C25—C26	1.483 (4)
C1—H1	0.9500	C26—C27	1.388 (4)

C2—C3	1.375 (4)	C27—C28	1.382 (4)
C2—H2	0.9500	C27—H27	0.9500
C3—C4	1.379 (4)	C28—C29	1.386 (4)
C3—H3	0.9500	C28—H28	0.9500
C4—C5	1.384 (4)	C29—C30	1.382 (4)
C4—H4	0.9500	C29—H29	0.9500
C5—C6	1.482 (4)	C30—C31	1.481 (4)
C6—C7	1.385 (4)	C31—C32	1.381 (4)
C7—C8	1.383 (4)	C32—C33	1.384 (4)
C7—H7	0.9500	C32—H32	0.9500
C8—C9	1.385 (4)	C33—C34	1.381 (4)
C8—H8	0.9500	C33—H33	0.9500
C9—C10	1.378 (4)	C34—C35	1.379 (4)
C9—H9	0.9500	C34—H34	0.9500
C10—C11	1.487 (4)	C35—H35	0.9500
C11—C12	1.383 (4)	Cl1—O2	1.409 (2)
C12—C13	1.383 (4)	Cl1—O3	1.418 (2)
C12—H12	0.9500	Cl1—O4	1.419 (2)
C13—C14	1.380 (4)	Cl1—O1	1.430 (2)
C13—H13	0.9500	Cl2—O12	1.429 (2)
C14—C15	1.383 (4)	Cl2—O11	1.434 (2)
C14—H14	0.9500	Cl2—O14	1.435 (2)
C15—H15	0.9500	Cl2—O13	1.436 (2)
N21—C21	1.345 (3)	O21—H21A	0.8400
N21—C25	1.354 (3)	O21—H21B	0.8398

N2—Ni1—N22	177.92 (8)	C14—C15—H15	118.7
N2—Ni1—N1	78.00 (8)	C21—N21—C25	118.6 (2)
N22—Ni1—N1	103.66 (8)	C21—N21—Ni1	126.84 (17)
N2—Ni1—N23	103.24 (8)	C25—N21—Ni1	114.52 (16)
N22—Ni1—N23	78.06 (8)	C26—N22—C30	120.8 (2)
N1—Ni1—N23	90.81 (8)	C26—N22—Ni1	120.04 (16)
N2—Ni1—N3	77.50 (8)	C30—N22—Ni1	119.17 (17)
N22—Ni1—N3	100.87 (8)	C35—N23—C31	118.5 (2)
N1—Ni1—N3	155.42 (8)	C35—N23—Ni1	127.55 (17)
N23—Ni1—N3	92.80 (8)	C31—N23—Ni1	113.88 (16)
N2—Ni1—N21	101.35 (8)	N21—C21—C22	122.5 (2)
N22—Ni1—N21	77.40 (8)	N21—C21—H21	118.7
N1—Ni1—N21	92.94 (8)	C22—C21—H21	118.7
N23—Ni1—N21	155.37 (8)	C23—C22—C21	119.1 (3)
N3—Ni1—N21	93.82 (8)	C23—C22—H22	120.4
C1—N1—C5	118.6 (2)	C21—C22—H22	120.4
C1—N1—Ni1	126.96 (17)	C22—C23—C24	119.1 (3)
C5—N1—Ni1	114.14 (16)	C22—C23—H23	120.4
C6—N2—C10	120.7 (2)	C24—C23—H23	120.4
C6—N2—Ni1	119.19 (17)	C25—C24—C23	118.9 (3)
C10—N2—Ni1	120.03 (17)	C25—C24—H24	120.5
C15—N3—C11	118.5 (2)	C23—C24—H24	120.5

C15—N3—Ni1	126.92 (18)	N21—C25—C24	121.7 (2)
C11—N3—Ni1	114.51 (16)	N21—C25—C26	114.6 (2)
N1—C1—C2	122.4 (2)	C24—C25—C26	123.7 (2)
N1—C1—H1	118.8	N22—C26—C27	121.0 (2)
C2—C1—H1	118.8	N22—C26—C25	113.4 (2)
C3—C2—C1	118.6 (2)	C27—C26—C25	125.6 (2)
C3—C2—H2	120.7	C28—C27—C26	118.5 (3)
C1—C2—H2	120.7	C28—C27—H27	120.8
C2—C3—C4	119.9 (3)	C26—C27—H27	120.8
C2—C3—H3	120.1	C27—C28—C29	120.4 (3)
C4—C3—H3	120.1	C27—C28—H28	119.8
C3—C4—C5	118.8 (3)	C29—C28—H28	119.8
C3—C4—H4	120.6	C30—C29—C28	118.4 (2)
C5—C4—H4	120.6	C30—C29—H29	120.8
N1—C5—C4	121.7 (2)	C28—C29—H29	120.8
N1—C5—C6	114.9 (2)	N22—C30—C29	121.1 (2)
C4—C5—C6	123.3 (2)	N22—C30—C31	113.5 (2)
N2—C6—C7	121.0 (2)	C29—C30—C31	125.4 (2)
N2—C6—C5	113.3 (2)	N23—C31—C32	121.8 (2)
C7—C6—C5	125.7 (2)	N23—C31—C30	115.2 (2)
C8—C7—C6	118.3 (3)	C32—C31—C30	123.0 (2)
C8—C7—H7	120.9	C31—C32—C33	119.1 (3)
C6—C7—H7	120.9	C31—C32—H32	120.5
C7—C8—C9	120.5 (3)	C33—C32—H32	120.5
C7—C8—H8	119.8	C34—C33—C32	119.2 (2)
C9—C8—H8	119.8	C34—C33—H33	120.4
C10—C9—C8	118.6 (2)	C32—C33—H33	120.4
C10—C9—H9	120.7	C35—C34—C33	118.9 (3)
C8—C9—H9	120.7	C35—C34—H34	120.5
N2—C10—C9	121.0 (2)	C33—C34—H34	120.5
N2—C10—C11	113.1 (2)	N23—C35—C34	122.4 (2)
C9—C10—C11	125.9 (2)	N23—C35—H35	118.8
N3—C11—C12	121.7 (2)	C34—C35—H35	118.8
N3—C11—C10	114.6 (2)	O2—Cl1—O3	109.1 (2)
C12—C11—C10	123.7 (2)	O2—Cl1—O4	110.48 (19)
C13—C12—C11	119.0 (3)	O3—Cl1—O4	108.34 (19)
C13—C12—H12	120.5	O2—Cl1—O1	109.14 (15)
C11—C12—H12	120.5	O3—Cl1—O1	109.75 (16)
C14—C13—C12	119.4 (2)	O4—Cl1—O1	110.00 (15)
C14—C13—H13	120.3	O12—Cl2—O11	109.68 (15)
C12—C13—H13	120.3	O12—Cl2—O14	110.85 (17)
C13—C14—C15	118.7 (3)	O11—Cl2—O14	109.55 (14)
C13—C14—H14	120.7	O12—Cl2—O13	108.86 (14)
C15—C14—H14	120.7	O11—Cl2—O13	108.73 (14)
N3—C15—C14	122.6 (3)	O14—Cl2—O13	109.14 (17)
N3—C15—H15	118.7	H21A—O21—H21B	103.2

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O21—H21*A*···O3	0.84	2.01	2.772 (5)	151
O21—H21*B*···O1^{i}	0.84	2.08	2.845 (5)	150
C1—H1···O2	0.95	2.58	3.490 (4)	162
C2—H2···O4	0.95	2.59	3.292 (4)	131
C4—H4···O12^{ii}	0.95	2.32	3.127 (3)	142
C9—H9···O13^{iii}	0.95	2.53	3.417 (3)	155
C12—H12···O13^{iii}	0.95	2.66	3.539 (3)	155
C15—H15···O3^{iv}	0.95	2.45	3.265 (4)	144
C15—H15···O21^{iv}	0.95	2.48	3.124 (5)	125
C21—H21···O11	0.95	2.52	3.255 (3)	134
C21—H21···O14	0.95	2.60	3.319 (4)	133
C24—H24···O2^{v}	0.95	2.65	3.575 (4)	166
C32—H32···O1^{vi}	0.95	2.39	3.243 (3)	149
C34—H34···O11^{vii}	0.95	2.52	3.095 (3)	119

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