11-15-2016

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Recommended Citation
Vasileiadou, E., Angaridis, P. A., Raptis, R. G. & Mathivathanan, L. (2016). IUCrData 1.

IUCrData

ISSN 2414-3146

Aquabis(2,2′-bipyridine-$\kappa^2 N,N'$)chloridonickel(II) chloride chloroform monosolvate hemihydrate

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Received 13 October 2016
Accepted 15 November 2016

Edited by M. Weil, Vienna University of Technology, Austria

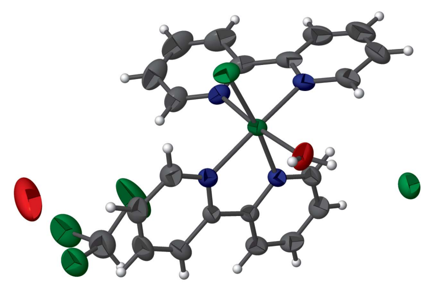
Keywords: crystal structure; nickel(II); 2,2′-bipyridine; octahedral coordination environment.

CCDC reference: 1517485

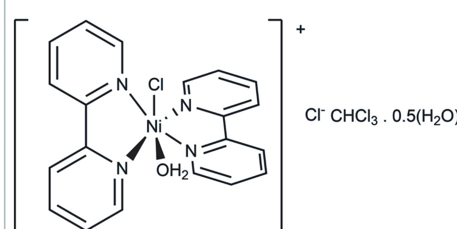
Structural data: full structural data are available from iucrdata.iucr.org

The title solvated salt, $[NiCl(C_{10}H_8N_2)_2(H_2O)]Cl\cdot CHCl_3\cdot 0.5H_2O$, contains a mononuclear Ni^{II} complex cation with 2,2′-bipyridine, chloride and aqua ligands forming a slightly distorted ClN_4O octahedral coordination set. The charge of the cation is balanced by a chloride anion. In the crystal, half a water molecule and a chloroform solvent molecule are present per formula unit. Individual components are held together by O—H⋯Cl hydrogen bonding and π–π interactions.

3D view



Chemical scheme



Structure description

The Ni^{II} cation has a distorted octahedral coordination environment with the chlorido and aqua ligands in a *cis* configuration relative to each other (Fig. 1). The two *N*,*N*′-bipyridine ligands are almost perpendicular to each other [dihedral angle 88.73 (16)°]. The Ni—N distances range between 2.059 (3) and 2.102 (3) Å, while the Ni—O(water) and Ni—Cl distances are 2.084 (3) and 2.418 (1) Å, respectively. Similar *cis*-$[M^{II}Cl(2,2'\text{-bipy})_2(H_2O)]^+$ cations are known for M = Mn (Chen *et al.*, 1995) and Cd (Lei & Li, 2011). A few $[NiLL')_2Cl(OHR)]^+$ complexes are known in the literature where LL' is a bidentate chelating N-donor ligand such as 2,2′-bipyridine or 1,10-phenanthroline. Examples with R = H or methyl were given by Brewer *et al.* (2003) and Chesnut *et al.* (1999). Interestingly, all except one adopt the *cis*-configuration. The *trans*-configuration between Cl and H_2O is known for a tetradentate bis-phenanthroline ligand, *viz*., 2,2′-bis-(1,10-phenanthroline) (Rice & Anderson, 2000), where steric hindrance presumably prevents a *cis* configuration. In one case, $[Ni(2,2'\text{-bipy})_2Cl(OH_2)]^+$ has been formed *in situ* by reacting $[Ni(2,2'\text{-bipy})_3]^{2+}$ with Cl^- and H_2O. The reaction was concentration-sensitive

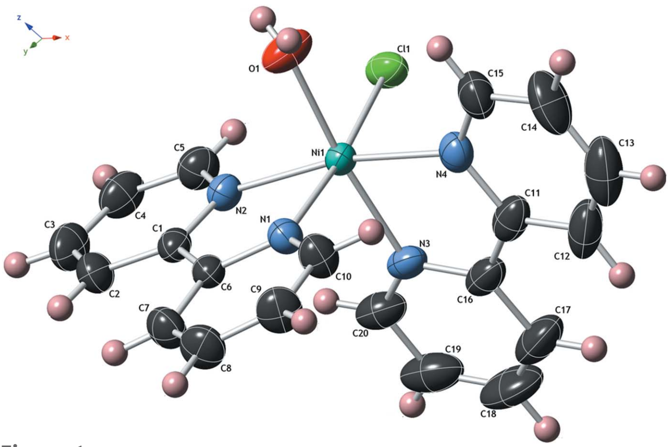


Figure 1
The molecular structure of the cation of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level.

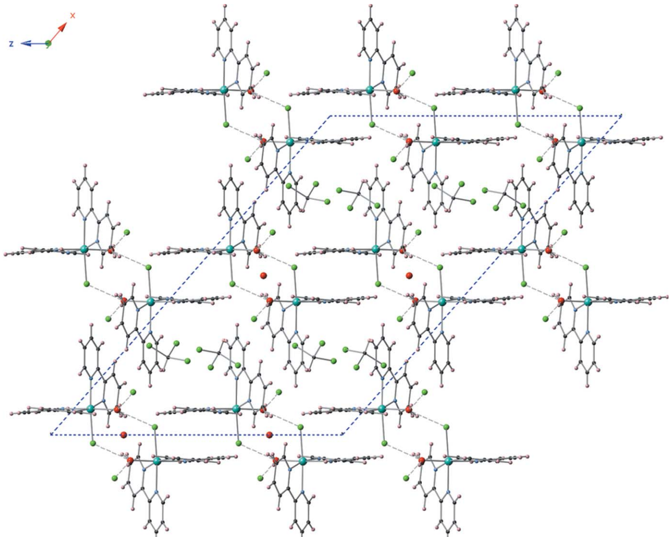


Figure 2
Packing diagram of the title compound, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

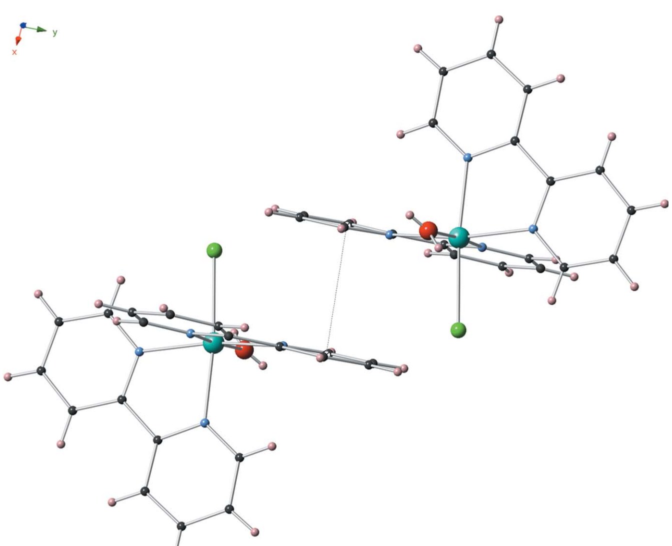


Figure 3
The intermolecular π–π interactions between the 2,2′-bipy ligands of adjacent complex cations in the title compound.

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
O1—H1$A$$\cdots$Cl5	0.87 (2)	2.25 (2)	3.115 (3)	171 (5)
O1—H1$B$$\cdots$Cl1^{i}	0.87 (2)	2.30 (2)	3.148 (3)	163 (4)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$[NiCl(C_{10}H_8N_2)_2(H_2O)]Cl\cdot CHCl_3\cdot 0.5H_2O$
M_r	588.36
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	300
a, b, c (Å)	29.1709 (14), 11.2898 (5), 20.0517 (10)
β (°)	131.163 (1)
V (Å^3)	4971.5 (4)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.34
Crystal size (mm)	0.14 × 0.07 × 0.06
Data collection	
Diffractometer	Bruker D8 Quest CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.692, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	42949, 5141, 3870
R_{int}	0.048
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.628
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.052, 0.140, 1.05
No. of reflections	5141
No. of parameters	300
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.56, −0.71

Computer programs: *APEX3* and *SAINT* (Bruker, 2015), *SHELXT2014* (Sheldrick, 2015), *ShelXle* (Hübschle *et al.*, 2011), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip *et al.*, 2010).

and the $[Ni(2,2'\text{-bipy})Cl(OH_2)]^+$ cation forms a three-dimensional hydrogen-bonded network with deprotonated benzene tetracarboxylic acid moieties (Sun *et al.*, 2010).

The asymmetric unit of the title compound also contains a $CHCl_3$ solvent molecule and a lattice water molecule located on a twofold rotation axis. The Ni-bound water (O1) molecule forms weak hydrogen bonds with the Cl^- counter-anion (Cl5) and the coordinating Cl (Cl1) atom from an adjacent molecule (Table 1, Fig. 2). Although the H atoms of the lattice water molecule could not be located, O$\cdots$Cl distances of 3.231 (3) Å to the counter-anion indicate likewise weak hydrogen bonding. π–π interactions between the pyridyl rings of parallel-stacked 2,2-bipy molecules [C11—C13 = 3.465 (6) Å] are also present in the crystal lattice (Fig. 3). It is worth noting that similar Cl-bridged hetero- and homo-binuclear compounds dominate $NiCl_2(LL')_2$-chemistry. One example of such heterobinuclear compound, $[Ni(2,2'\text{-bipy})_2(\mu\text{-Cl})_2CdI_2]$, has been reported (Chesnut *et al.*, 1999).

Synthesis and crystallization

The title compound was isolated when 2,2′-bipyridine was used as an auxiliary ligand for the intended preparation of a Ni-sulfonamide complex. A solution of $NiCl_2 \cdot 6H_2O$ (34.2 mg, 0.144 mmol) in 10 ml MeOH was added slowly to a solution of *N*,*N*-diphenyl-1,2-benzenesulfonamide (60 mg, 0.144 mmol) and 2,2′-bipyridine (25.5 mg, 0.163 mmol) in 8 ml MeOH and 2.2 eq. of $NHEt_2$, at room temperature. A precipitate formed after 10 min and the reaction was stirred for an additional three hours. The precipitate was filtered off the methanol solution; the yellow–green precipitate was collected and crystals were obtained by diffusion of diethyl ether vapor into a chloroform solution of the compound.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydrogen atoms of the lattice water molecule could not be modelled satisfactorily and were omitted from the refinement, but are included in the formula.

Acknowledgements

REU participant EV was supported by the NSF–REU Site Grant CHE1560375 to FIU.

References

Brewer, B., Brooks, N. R., Abdul-Halim, R. & Sykes, A. G. (2003). *J. Chem. Crystallogr.* **33**, 651–662.
Bruker (2015). *APEX3* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, X.-M., Shi, K.-L., Mak, T. C. W. & Luo, B.-S. (1995). *Acta Cryst.* C**51**, 358–361.
Chesnut, D. J., Haushalter, R. C. & Zubieta, J. (1999). *Inorg. Chim. Acta*, **292**, 41–51.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Hübschle, C. B., Sheldrick, G. M. & Dittrich, B. (2011). *J. Appl. Cryst.* **44**, 1281–1284.
Lei, Z.-H. & Li, X. (2011). *J. Coord. Chem.* **64**, 2450–2457.
Rice, C. R. & Anderson, K. M. (2000). *Polyhedron*, **19**, 495–498.
Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.
Sun, M.-L., Zhang, L., Lin, Q.-P., Zhang, J. & Yao, Y.-G. (2010). *Cryst. Growth Des.* **10**, 1464–1467.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2016). **1**, x161834 [https://doi.org/10.1107/S2414314616018344]

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Crystal data

$[NiCl(C_{10}H_8N_2)_2(H_2O)]Cl\cdot CHCl_3\cdot 0.5H_2O$
M_r = 588.36
Monoclinic, $C2/c$
a = 29.1709 (14) Å
b = 11.2898 (5) Å
c = 20.0517 (10) Å
β = 131.163 (1)°
V = 4971.5 (4) Å^3
Z = 8
$F(000)$ = 2392
D_x = 1.572 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9884 reflections
θ = 2.8–26.3°
μ = 1.34 mm^{-1}
T = 300 K
Trapezoid, blue
0.14 × 0.07 × 0.06 mm

Data collection

Bruker D8 Quest CMOS diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2015)
T_{min} = 0.692, T_{max} = 0.745
42949 measured reflections
5141 independent reflections
3870 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 26.5°, θ_{min} = 2.8°
$h = -36 \rightarrow 36$
$k = -14 \rightarrow 14$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.140
S = 1.05
5141 reflections
300 parameters
2 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 19.6746P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

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}
Ni1	0.41761 (2)	0.23752 (4)	0.55719 (3)	0.02914 (15)
Cl1	0.52576 (4)	0.27540 (10)	0.66856 (6)	0.0436 (3)
O1	0.42085 (15)	0.1520 (3)	0.65230 (19)	0.0481 (8)
N1	0.32221 (14)	0.2227 (3)	0.4674 (2)	0.0329 (7)
N2	0.39253 (14)	0.3961 (3)	0.5772 (2)	0.0329 (7)
N3	0.41843 (14)	0.3077 (3)	0.4617 (2)	0.0372 (8)
N4	0.42689 (14)	0.0861 (3)	0.5098 (2)	0.0387 (8)
C1	0.33271 (17)	0.4149 (3)	0.5258 (2)	0.0315 (8)
C2	0.3104 (2)	0.5186 (4)	0.5327 (3)	0.0444 (10)
H2	0.2687	0.5299	0.4980	0.053*
C3	0.3508 (2)	0.6044 (4)	0.5916 (3)	0.0532 (12)
H3	0.3367	0.6746	0.5968	0.064*
C4	0.4116 (2)	0.5858 (4)	0.6421 (3)	0.0494 (11)
H4	0.4395	0.6435	0.6816	0.059*
C5	0.4311 (2)	0.4808 (4)	0.6340 (3)	0.0437 (10)
H5	0.4727	0.4678	0.6694	0.052*
C6	0.29276 (16)	0.3194 (3)	0.4628 (2)	0.0313 (8)
C7	0.23009 (18)	0.3261 (4)	0.4031 (3)	0.0443 (10)
H7	0.2106	0.3939	0.3997	0.053*
C8	0.1965 (2)	0.2306 (4)	0.3481 (3)	0.0497 (11)
H8	0.1542	0.2334	0.3077	0.060*
C9	0.22615 (19)	0.1322 (4)	0.3538 (3)	0.0482 (11)
H9	0.2044	0.0667	0.3180	0.058*
C10	0.28837 (18)	0.1316 (4)	0.4131 (3)	0.0427 (10)
H10	0.3083	0.0648	0.4159	0.051*
C11	0.42223 (18)	0.1021 (4)	0.4384 (3)	0.0451 (11)
C12	0.4180 (2)	0.0055 (6)	0.3914 (4)	0.0672 (15)
H12	0.4138	0.0173	0.3417	0.081*
C13	0.4201 (3)	−0.1054 (6)	0.4185 (5)	0.0789 (18)
H13	0.4173	−0.1702	0.3874	0.095*
C14	0.4263 (2)	−0.1229 (5)	0.4919 (4)	0.0718 (16)
H14	0.4281	−0.1989	0.5113	0.086*
C15	0.4298 (2)	−0.0244 (4)	0.5363 (3)	0.0520 (11)
H15	0.4343	−0.0355	0.5863	0.062*
C16	0.42170 (18)	0.2267 (4)	0.4160 (3)	0.0450 (11)
C17	0.4276 (2)	0.2633 (6)	0.3550 (3)	0.0692 (16)
H17	0.4304	0.2072	0.3237	0.083*
C18	0.4294 (3)	0.3796 (7)	0.3418 (4)	0.0792 (19)
H18	0.4330	0.4040	0.3012	0.095*
C19	0.4259 (2)	0.4610 (6)	0.3881 (3)	0.0709 (16)
H19	0.4274	0.5415	0.3800	0.085*
C20	0.4200 (2)	0.4220 (4)	0.4472 (3)	0.0516 (11)
H20	0.4170	0.4779	0.4783	0.062*
H1A	0.402 (2)	0.088 (3)	0.646 (3)	0.062*
H1B	0.4432 (19)	0.182 (4)	0.7055 (18)	0.062*

Cl2	0.28738 (15)	0.61538 (18)	0.34457 (16)	0.1420 (10)
Cl3	0.23219 (9)	0.83353 (17)	0.25316 (11)	0.1017 (6)
Cl4	0.28069 (7)	0.80694 (13)	0.43133 (10)	0.0766 (4)
C21	0.2434 (3)	0.7354 (5)	0.3311 (4)	0.0801 (17)
H21	0.2039	0.7064	0.3091	0.096*
Cl5	0.36460 (5)	−0.08254 (10)	0.65231 (8)	0.0556 (3)
O2	0.5000	0.8219 (5)	0.7500	0.127 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0288 (3)	0.0340 (3)	0.0255 (2)	0.0032 (2)	0.0182 (2)	0.0000 (2)
Cl1	0.0293 (5)	0.0628 (7)	0.0319 (5)	0.0040 (4)	0.0172 (4)	0.0056 (5)
O1	0.064 (2)	0.0492 (18)	0.0373 (16)	−0.0183 (15)	0.0359 (16)	−0.0078 (14)
N1	0.0319 (16)	0.0323 (17)	0.0328 (16)	0.0022 (13)	0.0206 (15)	0.0001 (13)
N2	0.0335 (17)	0.0348 (17)	0.0306 (16)	−0.0012 (14)	0.0212 (15)	−0.0038 (13)
N3	0.0319 (17)	0.051 (2)	0.0259 (16)	−0.0028 (15)	0.0178 (15)	0.0018 (15)
N4	0.0329 (18)	0.044 (2)	0.0405 (19)	0.0055 (14)	0.0249 (16)	−0.0013 (15)
C1	0.038 (2)	0.0300 (19)	0.0320 (19)	0.0023 (16)	0.0256 (18)	0.0021 (15)
C2	0.047 (2)	0.041 (2)	0.046 (2)	0.0100 (19)	0.031 (2)	0.0027 (19)
C3	0.075 (3)	0.036 (2)	0.058 (3)	0.004 (2)	0.048 (3)	−0.005 (2)
C4	0.063 (3)	0.041 (2)	0.046 (3)	−0.013 (2)	0.037 (2)	−0.014 (2)
C5	0.043 (2)	0.046 (2)	0.040 (2)	−0.0075 (19)	0.026 (2)	−0.0091 (19)
C6	0.034 (2)	0.034 (2)	0.0308 (19)	0.0052 (16)	0.0230 (17)	0.0041 (15)
C7	0.034 (2)	0.048 (3)	0.046 (2)	0.0063 (19)	0.024 (2)	0.002 (2)
C8	0.029 (2)	0.063 (3)	0.045 (2)	−0.002 (2)	0.019 (2)	−0.003 (2)
C9	0.038 (2)	0.048 (3)	0.045 (2)	−0.012 (2)	0.022 (2)	−0.013 (2)
C10	0.039 (2)	0.037 (2)	0.047 (2)	−0.0012 (18)	0.026 (2)	−0.0074 (19)
C11	0.033 (2)	0.068 (3)	0.038 (2)	0.001 (2)	0.025 (2)	−0.010 (2)
C12	0.063 (3)	0.085 (4)	0.070 (3)	−0.007 (3)	0.051 (3)	−0.031 (3)
C13	0.075 (4)	0.075 (4)	0.104 (5)	−0.002 (3)	0.066 (4)	−0.036 (4)
C14	0.064 (3)	0.049 (3)	0.108 (5)	0.011 (2)	0.059 (4)	−0.008 (3)
C15	0.050 (3)	0.051 (3)	0.061 (3)	0.011 (2)	0.039 (2)	0.000 (2)
C16	0.032 (2)	0.075 (3)	0.028 (2)	0.000 (2)	0.0200 (18)	−0.002 (2)
C17	0.066 (3)	0.112 (5)	0.044 (3)	−0.004 (3)	0.043 (3)	−0.006 (3)
C18	0.076 (4)	0.123 (6)	0.049 (3)	−0.010 (4)	0.045 (3)	0.017 (3)
C19	0.065 (3)	0.086 (4)	0.048 (3)	−0.017 (3)	0.032 (3)	0.015 (3)
C20	0.051 (3)	0.061 (3)	0.037 (2)	−0.008 (2)	0.027 (2)	0.004 (2)
Cl2	0.261 (3)	0.0811 (13)	0.1370 (18)	0.0660 (16)	0.154 (2)	0.0285 (12)
Cl3	0.1164 (14)	0.0971 (13)	0.0744 (10)	0.0275 (11)	0.0554 (11)	0.0225 (9)
Cl4	0.0888 (10)	0.0663 (9)	0.0773 (9)	0.0039 (8)	0.0557 (9)	−0.0023 (7)
C21	0.079 (4)	0.073 (4)	0.082 (4)	0.000 (3)	0.050 (4)	−0.003 (3)
Cl5	0.0589 (7)	0.0393 (6)	0.0658 (7)	0.0008 (5)	0.0398 (6)	0.0006 (5)
O2	0.064 (4)	0.053 (4)	0.170 (7)	0.000	0.036 (4)	0.000

Geometric parameters (Å, º)

Ni1—N4	2.059 (3)	C7—H7	0.9300
Ni1—N2	2.071 (3)	C8—C9	1.365 (6)
Ni1—O1	2.084 (3)	C8—H8	0.9300
Ni1—N3	2.087 (3)	C9—C10	1.366 (6)
Ni1—N1	2.102 (3)	C9—H9	0.9300
Ni1—Cl1	2.4183 (11)	C10—H10	0.9300
O1—H1A	0.872 (19)	C11—C12	1.393 (6)
O1—H1B	0.874 (19)	C11—C16	1.473 (7)
N1—C10	1.343 (5)	C12—C13	1.351 (9)
N1—C6	1.355 (5)	C12—H12	0.9300
N2—C1	1.336 (5)	C13—C14	1.374 (8)
N2—C5	1.338 (5)	C13—H13	0.9300
N3—C20	1.330 (6)	C14—C15	1.385 (7)
N3—C16	1.341 (5)	C14—H14	0.9300
N4—C15	1.337 (6)	C15—H15	0.9300
N4—C11	1.357 (5)	C16—C17	1.407 (6)
C1—C2	1.390 (5)	C17—C18	1.347 (8)
C1—C6	1.476 (5)	C17—H17	0.9300
C2—C3	1.374 (6)	C18—C19	1.356 (9)
C2—H2	0.9300	C18—H18	0.9300
C3—C4	1.361 (7)	C19—C20	1.378 (7)
C3—H3	0.9300	C19—H19	0.9300
C4—C5	1.370 (6)	C20—H20	0.9300
C4—H4	0.9300	Cl2—C21	1.760 (6)
C5—H5	0.9300	Cl3—C21	1.761 (7)
C6—C7	1.378 (5)	Cl4—C21	1.731 (6)
C7—C8	1.384 (6)	C21—H21	0.9800
N4—Ni1—N2	167.95 (13)	C6—C7—C8	119.2 (4)
N4—Ni1—O1	95.59 (13)	C6—C7—H7	120.4
N2—Ni1—O1	91.87 (12)	C8—C7—H7	120.4
N4—Ni1—N3	78.85 (14)	C9—C8—C7	119.4 (4)
N2—Ni1—N3	93.92 (13)	C9—C8—H8	120.3
O1—Ni1—N3	174.13 (13)	C7—C8—H8	120.3
N4—Ni1—N1	92.32 (12)	C8—C9—C10	118.8 (4)
N2—Ni1—N1	78.32 (12)	C8—C9—H9	120.6
O1—Ni1—N1	89.09 (12)	C10—C9—H9	120.6
N3—Ni1—N1	93.00 (12)	N1—C10—C9	123.2 (4)
N4—Ni1—Cl1	94.88 (9)	N1—C10—H10	118.4
N2—Ni1—Cl1	94.63 (9)	C9—C10—H10	118.4
O1—Ni1—Cl1	89.50 (9)	N4—C11—C12	120.8 (5)
N3—Ni1—Cl1	89.10 (9)	N4—C11—C16	114.9 (4)
N1—Ni1—Cl1	172.76 (9)	C12—C11—C16	124.2 (4)
Ni1—O1—H1A	127 (3)	C13—C12—C11	119.5 (5)
Ni1—O1—H1B	119 (3)	C13—C12—H12	120.2
H1A—O1—H1B	114 (5)	C11—C12—H12	120.2

C10—N1—C6	117.9 (3)	C12—C13—C14	120.2 (5)
C10—N1—Ni1	127.4 (3)	C12—C13—H13	119.9
C6—N1—Ni1	114.6 (2)	C14—C13—H13	119.9
C1—N2—C5	118.9 (3)	C13—C14—C15	118.4 (6)
C1—N2—Ni1	115.9 (2)	C13—C14—H14	120.8
C5—N2—Ni1	125.2 (3)	C15—C14—H14	120.8
C20—N3—C16	119.0 (4)	N4—C15—C14	122.3 (5)
C20—N3—Ni1	126.2 (3)	N4—C15—H15	118.8
C16—N3—Ni1	114.6 (3)	C14—C15—H15	118.8
C15—N4—C11	118.7 (4)	N3—C16—C17	119.9 (5)
C15—N4—Ni1	125.7 (3)	N3—C16—C11	115.7 (4)
C11—N4—Ni1	115.1 (3)	C17—C16—C11	124.3 (4)
N2—C1—C2	121.1 (4)	C18—C17—C16	120.0 (5)
N2—C1—C6	116.0 (3)	C18—C17—H17	120.0
C2—C1—C6	122.9 (3)	C16—C17—H17	120.0
C3—C2—C1	119.1 (4)	C17—C18—C19	119.8 (5)
C3—C2—H2	120.4	C17—C18—H18	120.1
C1—C2—H2	120.4	C19—C18—H18	120.1
C4—C3—C2	119.4 (4)	C18—C19—C20	118.7 (6)
C4—C3—H3	120.3	C18—C19—H19	120.6
C2—C3—H3	120.3	C20—C19—H19	120.6
C3—C4—C5	119.0 (4)	N3—C20—C19	122.6 (5)
C3—C4—H4	120.5	N3—C20—H20	118.7
C5—C4—H4	120.5	C19—C20—H20	118.7
N2—C5—C4	122.5 (4)	Cl4—C21—Cl2	110.1 (4)
N2—C5—H5	118.7	Cl4—C21—Cl3	110.2 (3)
C4—C5—H5	118.7	Cl2—C21—Cl3	108.2 (4)
N1—C6—C7	121.5 (4)	Cl4—C21—H21	109.5
N1—C6—C1	115.0 (3)	Cl2—C21—H21	109.5
C7—C6—C1	123.5 (3)	Cl3—C21—H21	109.5

C5—N2—C1—C2	−2.0 (5)	C15—N4—C11—C12	2.6 (6)
Ni1—N2—C1—C2	−179.4 (3)	Ni1—N4—C11—C12	−169.8 (3)
C5—N2—C1—C6	178.2 (3)	C15—N4—C11—C16	−177.7 (4)
Ni1—N2—C1—C6	0.8 (4)	Ni1—N4—C11—C16	9.9 (4)
N2—C1—C2—C3	2.0 (6)	N4—C11—C12—C13	−1.7 (7)
C6—C1—C2—C3	−178.3 (4)	C16—C11—C12—C13	178.7 (5)
C1—C2—C3—C4	−0.4 (7)	C11—C12—C13—C14	0.1 (8)
C2—C3—C4—C5	−1.1 (7)	C12—C13—C14—C15	0.5 (8)
C1—N2—C5—C4	0.5 (6)	C11—N4—C15—C14	−2.1 (6)
Ni1—N2—C5—C4	177.7 (3)	Ni1—N4—C15—C14	169.5 (4)
C3—C4—C5—N2	1.1 (7)	C13—C14—C15—N4	0.5 (8)
C10—N1—C6—C7	−1.6 (5)	C20—N3—C16—C17	1.0 (6)
Ni1—N1—C6—C7	175.9 (3)	Ni1—N3—C16—C17	−174.8 (3)
C10—N1—C6—C1	178.5 (3)	C20—N3—C16—C11	177.6 (4)
Ni1—N1—C6—C1	−4.0 (4)	Ni1—N3—C16—C11	1.8 (4)
N2—C1—C6—N1	2.2 (5)	N4—C11—C16—N3	−7.7 (5)
C2—C1—C6—N1	−177.6 (4)	C12—C11—C16—N3	171.9 (4)

N2—C1—C6—C7	−177.7 (4)	N4—C11—C16—C17	168.7 (4)
C2—C1—C6—C7	2.5 (6)	C12—C11—C16—C17	−11.7 (7)
N1—C6—C7—C8	1.7 (6)	N3—C16—C17—C18	−0.7 (8)
C1—C6—C7—C8	−178.4 (4)	C11—C16—C17—C18	−177.0 (5)
C6—C7—C8—C9	−0.4 (7)	C16—C17—C18—C19	0.5 (9)
C7—C8—C9—C10	−0.9 (7)	C17—C18—C19—C20	−0.6 (9)
C6—N1—C10—C9	0.2 (6)	C16—N3—C20—C19	−1.1 (6)
Ni1—N1—C10—C9	−177.0 (3)	Ni1—N3—C20—C19	174.1 (3)
C8—C9—C10—N1	1.1 (7)	C18—C19—C20—N3	0.9 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···Cl5	0.87 (2)	2.25 (2)	3.115 (3)	171 (5)
O1—H1*B*···Cl1^{i}	0.87 (2)	2.30 (2)	3.148 (3)	163 (4)

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