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Bis(2,2′-bipyridine)[5,5′-iminobis(tetrazolato)]nickel(II) hexahydrate

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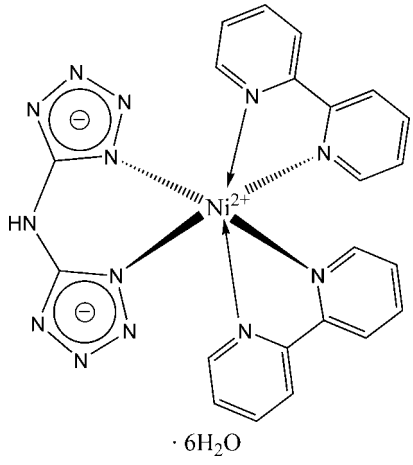
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.028; wR factor = 0.075; data-to-parameter ratio = 13.1.

The title complex, $[Ni(C_2HN_9)(C_{10}H_8N_2)_2]\cdot 6H_2O$, was prepared under hydrothermal reaction conditions. The asymmetric unit contains the nickel complex and six solvent water molecules. The Ni^{II} ion is coordinated in a distorted octahedral geometry defined by four N atoms from two 2,2′-bipyridine ligands and two N atoms from the 5,5′-iminobis(tetrazolate) (BTA^{2-}) anions. In the crystal structure, an extensive range of O—H···N and O—H···O hydrogen bonds links the complex and the water molecules into a three-dimensional network.

Related literature

Other complexes of the *N*,*N*-bis[1(2*H*)-tetrazol-5-yl]amine ligand are rare; for a related copper(II) complex, see: Friedrich *et al.* (2005).



Experimental

Crystal data

$[Ni(C_2HN_9)(C_{10}H_8N_2)_2]\cdot 6H_2O$
M_r = 630.27
Monoclinic, $P2_1/n$
a = 13.3435 (3) Å
b = 13.3741 (3) Å
c = 15.9408 (3) Å
β = 96.166 (1)°
V = 2828.29 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.75 mm^{-1}
T = 296 (2) K
0.25 × 0.25 × 0.13 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.830, T_{max} = 0.907
6514 measured reflections
6503 independent reflections
5453 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.075
S = 1.03
6503 reflections
496 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N6	2.0624 (12)	Ni1—N13	2.0941 (13)
Ni1—N10	2.0754 (13)	Ni1—N12	2.0968 (12)
Ni1—N1	2.0800 (12)	Ni1—N11	2.1070 (12)
N6—Ni1—N1	83.67 (5)	N10—Ni1—N12	170.80 (5)
N10—Ni1—N1	96.27 (5)	N13—Ni1—N12	78.39 (5)
N6—Ni1—N13	171.13 (5)	N1—Ni1—N11	174.71 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H1···O2^{i}	0.794 (19)	1.99 (2)	2.770 (2)	167.7 (19)
O1—H19···N8^{ii}	0.81 (3)	2.09 (3)	2.899 (2)	174 (3)
O1—H18···N9^{iii}	0.86 (3)	2.02 (3)	2.864 (2)	165 (3)
O2—H20···N4^{iv}	0.80 (3)	2.08 (3)	2.853 (2)	161 (3)
O2—H21···O4	0.84 (3)	1.90 (3)	2.724 (3)	168 (3)
O3—H23···N2^{v}	0.75 (3)	2.26 (3)	2.981 (2)	163 (3)
O3—H22···O1	0.82 (3)	2.08 (3)	2.885 (3)	168 (3)
O4—H25···O6^{v}	0.83 (3)	2.03 (3)	2.849 (3)	171 (3)
O4—H24···O3	0.82 (3)	2.12 (3)	2.930 (3)	167 (3)

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

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2005); 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: SJ2411).

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supplementary materials

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Bis(2,2'-bipyridine)[5,5'-iminobis(tetrazolato)]nickel(II) hexahydrate

X.-P. Liu and C. Zhang

Comment

Metal complexes of *N*,*N*-bis-(1(2)*H*-tetrazol-5-yl)-amine have not been extensively examined even though the deprotonated ligand contains nine potentially electron-donating nitrogen atoms (Friedrich *et al.*, 2005). The asymmetric unit of the title compound (I) comprises a Ni(II) cation, two 2,2'-bipyridine and one $HBTA^{2-}$ ligands and six solvent water molecules. Each Ni^{II} atom is coordinated to six N atoms from two 2,2'-bipyridine and one $HBTA^{2-}$ ligands to form a distorted mononuclear octahedral complex. (Table 1 and Fig. 1). In the crystal structure, an extensive range of O—H···N and O—H···O hydrogen bonds link the complex and water molecules into a three dimensional network, Table 2, Fig. 2.

Experimental

A mixture of $Ni(Cl)_2{\cdot}6H_2O$ (0.024 g, 0.1 mmol), bistetrazolylimine (0.031 g, 0.02 mmol) and water (20 ml) was heated in a 25 ml Teflon-lined autoclave at 433 K for 3 d, followed by slow cooling to room temperature. The resulting mixture was filtered, washed with 95% methanol and red crystals were collected and dried in air. Elemental analysis, calcd (%) for $C_{22}H_{29}N_{13}Ni_1O_6$: C 41.90, H 4.60, N 28.89; found(%): C 41.66, H 4.93, N 28.71.

Refinement

All hydrogen atoms were located in difference Fourier maps and freely refined with isotropic displacement parameters.

Figures

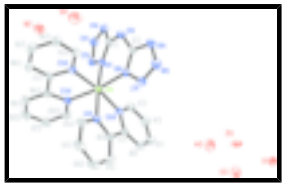

Fig. 1. The asymmetric unit of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

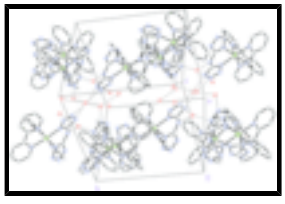

Fig. 2. The packing of the complex, showing a three-dimensional network connected by O—H···N and O—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

Bis(2,2'-bipyridine)[5,5'-iminobis(tetrazolato)]nickel(II) hexahydrate

Crystal data

$[Ni(C_2H_1N_9)(C_{10}H_8N_2)_2]\cdot 6H_2O$	F_{000} = 1312.0
M_r = 630.27	D_x = 1.480 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 5453 reflections
a = 13.3435 (3) Å	θ = 1.0–27.5º
b = 13.3741 (3) Å	μ = 0.75 mm^{-1}
c = 15.9408 (3) Å	T = 296 (2) K
β = 96.166 (1)º	Block, red
V = 2828.29 (10) Å^3	0.25 × 0.25 × 0.13 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	6503 independent reflections
Radiation source: fine-focus sealed tube	5453 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 296(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −17→16
T_{min} = 0.830, T_{max} = 0.907	k = −16→17
6514 measured reflections	l = −20→20

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)]$ = 0.028	$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.8759P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.075	$(\Delta/\sigma)_{max}$ = 0.033
S = 1.03	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
6503 reflections	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
496 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0018
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Ni1	0.721520 (13)	0.120775 (13)	0.874767 (11)	0.02942 (7)
C1	0.59648 (11)	0.30488 (11)	0.90861 (9)	0.0327 (3)
N4	0.50270 (10)	0.33969 (10)	0.90652 (9)	0.0394 (3)
N1	0.60038 (9)	0.21948 (9)	0.86584 (8)	0.0331 (3)
N5	0.67785 (10)	0.35362 (11)	0.94830 (10)	0.0422 (3)
N6	0.80563 (9)	0.24861 (9)	0.90063 (8)	0.0331 (3)
N3	0.44671 (10)	0.27279 (11)	0.85844 (9)	0.0422 (3)
N7	0.90583 (10)	0.26122 (11)	0.89697 (9)	0.0426 (3)
N2	0.50342 (9)	0.20166 (10)	0.83404 (8)	0.0390 (3)
N8	0.93118 (11)	0.34970 (12)	0.92545 (11)	0.0530 (4)
N9	0.85003 (11)	0.39895 (11)	0.94812 (10)	0.0487 (4)
C2	0.77503 (11)	0.33450 (11)	0.93208 (9)	0.0337 (3)
N10	0.74488 (10)	0.12590 (9)	0.74820 (8)	0.0385 (3)
N11	0.84590 (9)	0.02420 (9)	0.87217 (8)	0.0349 (3)
C8	0.87850 (11)	0.01380 (12)	0.79555 (10)	0.0376 (3)
C7	0.82656 (12)	0.07651 (12)	0.72765 (10)	0.0385 (3)
N12	0.70809 (9)	0.09258 (9)	1.00247 (7)	0.0332 (3)
C11	0.96054 (14)	−0.10370 (14)	0.92476 (14)	0.0516 (4)
C3	0.69266 (16)	0.18066 (15)	0.68884 (11)	0.0541 (5)
C12	0.88732 (13)	−0.03314 (13)	0.93510 (11)	0.0429 (4)
C9	0.95315 (13)	−0.05460 (15)	0.78158 (13)	0.0519 (5)
C6	0.85729 (16)	0.08335 (16)	0.64746 (12)	0.0543 (5)
C10	0.99352 (14)	−0.11430 (15)	0.84671 (14)	0.0572 (5)
C4	0.71786 (19)	0.18806 (17)	0.60757 (12)	0.0652 (6)
C5	0.8023 (2)	0.13933 (17)	0.58733 (12)	0.0657 (6)
N13	0.62922 (10)	−0.00624 (9)	0.86724 (8)	0.0361 (3)
C14	0.74236 (14)	0.11884 (14)	1.15098 (11)	0.0474 (4)
C18	0.61425 (11)	−0.04722 (11)	0.94200 (10)	0.0370 (3)
C13	0.74823 (13)	0.14660 (13)	1.06856 (10)	0.0399 (3)
C17	0.65970 (11)	0.00724 (11)	1.01771 (10)	0.0354 (3)
C22	0.58868 (14)	−0.05149 (14)	0.79670 (12)	0.0487 (4)
C16	0.65403 (15)	−0.02582 (15)	1.09968 (12)	0.0514 (4)
C15	0.69570 (16)	0.03107 (16)	1.16643 (12)	0.0551 (5)

C19	0.55832 (15)	−0.13399 (13)	0.94686 (13)	0.0504 (4)
C20	0.51727 (16)	−0.17936 (15)	0.87395 (14)	0.0582 (5)
C21	0.53251 (16)	−0.13798 (15)	0.79769 (14)	0.0587 (5)
O1	0.88828 (12)	0.53330 (12)	0.08779 (10)	0.0591 (4)
O2	0.62370 (14)	0.49551 (13)	0.06176 (10)	0.0710 (5)
O3	0.86502 (14)	0.35155 (15)	0.18119 (12)	0.0705 (4)
O6	0.09069 (16)	0.14894 (15)	0.86339 (14)	0.0818 (5)
O4	0.64923 (18)	0.37076 (15)	0.19747 (13)	0.0818 (5)
O5	0.22910 (15)	0.30002 (18)	0.87163 (17)	0.0998 (8)
H1	0.6674 (14)	0.4000 (15)	0.9771 (12)	0.046 (5)*
H19	0.941 (2)	0.562 (2)	0.0823 (17)	0.089 (9)*
H18	0.876 (2)	0.503 (2)	0.0403 (18)	0.099 (10)*
H20	0.578 (2)	0.534 (2)	0.0649 (17)	0.089 (9)*
H21	0.625 (2)	0.461 (2)	0.1060 (18)	0.091 (9)*
H23	0.906 (2)	0.349 (2)	0.2174 (19)	0.091 (11)*
H22	0.880 (2)	0.404 (2)	0.1587 (18)	0.095 (10)*
H25	0.630 (2)	0.371 (2)	0.245 (2)	0.094 (11)*
H24	0.710 (2)	0.359 (2)	0.1999 (19)	0.101 (12)*
H26	0.283 (2)	0.297 (2)	0.8633 (17)	0.091 (10)*
H28	0.133 (2)	0.193 (2)	0.8757 (16)	0.082 (9)*
H2	0.6373 (16)	0.2133 (16)	0.7053 (12)	0.061 (6)*
H3	0.6782 (16)	0.2259 (17)	0.5682 (14)	0.067 (6)*
H4	0.8253 (18)	0.1469 (17)	0.5331 (15)	0.077 (7)*
H5	0.9156 (16)	0.0511 (16)	0.6364 (13)	0.066 (6)*
H6	0.9746 (14)	−0.0575 (15)	0.7262 (13)	0.056 (5)*
H7	1.0420 (16)	−0.1609 (16)	0.8382 (12)	0.062 (6)*
H8	0.9863 (16)	−0.1419 (16)	0.9707 (13)	0.059 (6)*
H9	0.8632 (13)	−0.0252 (13)	0.9892 (11)	0.041 (5)*
H10	0.7829 (13)	0.2045 (14)	1.0568 (11)	0.044 (5)*
H11	0.7717 (15)	0.1569 (15)	1.1940 (12)	0.053 (5)*
H13	0.6198 (16)	−0.0869 (17)	1.1047 (13)	0.060 (6)*
H12	0.6918 (16)	0.0081 (16)	1.2213 (14)	0.069 (6)*
H17	0.5993 (14)	−0.0226 (14)	0.7453 (12)	0.045 (5)*
H14	0.5487 (15)	−0.1618 (16)	0.9997 (13)	0.064 (6)*
H16	0.5067 (17)	−0.1663 (17)	0.7484 (14)	0.070 (6)*
H15	0.4802 (16)	−0.2377 (17)	0.8765 (12)	0.063 (6)*
H29	0.035 (3)	0.178 (3)	0.867 (2)	0.140 (14)*
H27	0.211 (2)	0.354 (2)	0.8758 (18)	0.085 (10)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02916 (11)	0.02725 (10)	0.03302 (10)	0.00047 (7)	0.00869 (7)	−0.00243 (7)
C1	0.0313 (7)	0.0298 (7)	0.0387 (7)	0.0022 (6)	0.0115 (6)	0.0013 (6)
N4	0.0325 (7)	0.0364 (7)	0.0510 (7)	0.0043 (6)	0.0117 (6)	0.0013 (6)
N1	0.0278 (6)	0.0312 (6)	0.0409 (6)	−0.0003 (5)	0.0068 (5)	−0.0021 (5)
N5	0.0341 (7)	0.0356 (7)	0.0580 (8)	0.0006 (6)	0.0104 (6)	−0.0175 (6)
N6	0.0273 (6)	0.0320 (6)	0.0414 (6)	−0.0015 (5)	0.0105 (5)	−0.0013 (5)

N3	0.0302 (7)	0.0423 (8)	0.0548 (8)	0.0015 (6)	0.0082 (6)	0.0030 (6)
N7	0.0288 (7)	0.0444 (8)	0.0563 (8)	−0.0037 (6)	0.0116 (6)	−0.0054 (6)
N2	0.0300 (7)	0.0394 (7)	0.0479 (7)	−0.0021 (5)	0.0052 (5)	−0.0005 (6)
N8	0.0349 (8)	0.0490 (9)	0.0764 (11)	−0.0098 (6)	0.0116 (7)	−0.0122 (8)
N9	0.0372 (8)	0.0396 (8)	0.0695 (10)	−0.0077 (6)	0.0076 (7)	−0.0122 (7)
C2	0.0318 (7)	0.0307 (7)	0.0391 (7)	−0.0024 (6)	0.0062 (6)	−0.0025 (6)
N10	0.0425 (7)	0.0379 (7)	0.0365 (6)	0.0035 (6)	0.0104 (5)	−0.0026 (5)
N11	0.0317 (6)	0.0312 (6)	0.0421 (7)	0.0007 (5)	0.0056 (5)	−0.0058 (5)
C8	0.0315 (8)	0.0366 (8)	0.0455 (8)	−0.0032 (6)	0.0078 (6)	−0.0137 (6)
C7	0.0403 (8)	0.0355 (8)	0.0413 (8)	−0.0045 (7)	0.0123 (6)	−0.0109 (6)
N12	0.0324 (6)	0.0323 (6)	0.0358 (6)	−0.0006 (5)	0.0079 (5)	−0.0006 (5)
C11	0.0412 (10)	0.0439 (10)	0.0665 (12)	0.0080 (8)	−0.0095 (9)	−0.0068 (9)
C3	0.0660 (13)	0.0539 (11)	0.0440 (9)	0.0177 (10)	0.0129 (9)	0.0037 (8)
C12	0.0371 (9)	0.0402 (9)	0.0507 (9)	0.0039 (7)	0.0017 (7)	−0.0030 (7)
C9	0.0394 (9)	0.0593 (11)	0.0577 (11)	0.0069 (8)	0.0084 (8)	−0.0232 (9)
C6	0.0604 (12)	0.0584 (11)	0.0481 (10)	−0.0001 (10)	0.0239 (9)	−0.0107 (9)
C10	0.0385 (10)	0.0538 (11)	0.0772 (13)	0.0144 (8)	−0.0044 (9)	−0.0243 (10)
C4	0.0919 (17)	0.0631 (13)	0.0416 (10)	0.0147 (12)	0.0109 (10)	0.0073 (9)
C5	0.0970 (18)	0.0636 (13)	0.0410 (10)	0.0010 (12)	0.0279 (11)	−0.0031 (9)
N13	0.0343 (7)	0.0322 (6)	0.0421 (7)	−0.0016 (5)	0.0060 (5)	−0.0054 (5)
C14	0.0502 (10)	0.0561 (11)	0.0358 (8)	−0.0015 (8)	0.0040 (7)	−0.0024 (8)
C18	0.0318 (8)	0.0304 (8)	0.0497 (9)	0.0006 (6)	0.0086 (6)	−0.0002 (6)
C13	0.0438 (9)	0.0386 (8)	0.0378 (8)	−0.0037 (7)	0.0058 (7)	−0.0018 (6)
C17	0.0319 (7)	0.0324 (8)	0.0430 (8)	0.0005 (6)	0.0089 (6)	0.0029 (6)
C22	0.0500 (10)	0.0481 (10)	0.0485 (10)	−0.0061 (8)	0.0068 (8)	−0.0110 (8)
C16	0.0569 (11)	0.0462 (10)	0.0521 (10)	−0.0092 (9)	0.0103 (8)	0.0127 (8)
C15	0.0621 (12)	0.0648 (12)	0.0390 (9)	−0.0037 (10)	0.0081 (8)	0.0115 (8)
C19	0.0504 (11)	0.0377 (9)	0.0639 (11)	−0.0091 (8)	0.0094 (9)	0.0029 (8)
C20	0.0549 (11)	0.0380 (10)	0.0821 (14)	−0.0139 (9)	0.0092 (10)	−0.0080 (9)
C21	0.0547 (12)	0.0534 (11)	0.0675 (13)	−0.0126 (9)	0.0043 (10)	−0.0251 (10)
O1	0.0485 (8)	0.0625 (9)	0.0674 (9)	−0.0159 (7)	0.0111 (7)	−0.0119 (7)
O2	0.0947 (13)	0.0624 (10)	0.0601 (9)	0.0376 (9)	0.0285 (8)	0.0006 (7)
O3	0.0740 (11)	0.0698 (11)	0.0654 (10)	−0.0050 (9)	−0.0035 (9)	0.0078 (8)
O6	0.0624 (11)	0.0695 (11)	0.1171 (15)	−0.0019 (10)	0.0259 (11)	−0.0158 (10)
O4	0.0784 (14)	0.0903 (13)	0.0747 (12)	−0.0025 (10)	−0.0016 (10)	0.0195 (10)
O5	0.0458 (10)	0.0870 (15)	0.169 (2)	−0.0099 (10)	0.0244 (12)	−0.0504 (14)

Geometric parameters (Å, °)

Ni1—N6	2.0624 (12)	C6—H5	0.92 (2)
Ni1—N10	2.0754 (13)	C10—H7	0.92 (2)
Ni1—N1	2.0800 (12)	C4—C5	1.369 (3)
Ni1—N13	2.0941 (13)	C4—H3	0.93 (2)
Ni1—N12	2.0968 (12)	C5—H4	0.95 (2)
Ni1—N11	2.1070 (12)	N13—C22	1.339 (2)
C1—N4	1.3322 (19)	N13—C18	1.346 (2)
C1—N1	1.3340 (18)	C14—C15	1.363 (3)
C1—N5	1.363 (2)	C14—C13	1.376 (2)
N4—N3	1.3502 (19)	C14—H11	0.91 (2)

N1—N2	1.3589 (17)	C18—C19	1.386 (2)
N5—C2	1.3732 (19)	C18—C17	1.483 (2)
N5—H1	0.794 (19)	C13—H10	0.931 (19)
N6—C2	1.3348 (19)	C17—C16	1.389 (2)
N6—N7	1.3551 (17)	C22—C21	1.379 (3)
N3—N2	1.3007 (19)	C22—H17	0.930 (18)
N7—N8	1.299 (2)	C16—C15	1.376 (3)
N8—N9	1.349 (2)	C16—H13	0.94 (2)
N9—C2	1.325 (2)	C15—H12	0.93 (2)
N10—C3	1.333 (2)	C19—C20	1.372 (3)
N10—C7	1.345 (2)	C19—H14	0.94 (2)
N11—C12	1.334 (2)	C20—C21	1.371 (3)
N11—C8	1.3470 (19)	C20—H15	0.93 (2)
C8—C9	1.388 (2)	C21—H16	0.91 (2)
C8—C7	1.481 (2)	O1—H19	0.81 (3)
C7—C6	1.387 (2)	O1—H18	0.86 (3)
N12—C13	1.341 (2)	O2—H20	0.80 (3)
N12—C17	1.3462 (19)	O2—H21	0.84 (3)
C11—C10	1.371 (3)	O3—H23	0.75 (3)
C11—C12	1.381 (2)	O3—H22	0.82 (3)
C11—H8	0.93 (2)	O6—H28	0.82 (3)
C3—C4	1.377 (3)	O6—H29	0.85 (4)
C3—H2	0.92 (2)	O4—H25	0.83 (3)
C12—H9	0.959 (17)	O4—H24	0.82 (3)
C9—C10	1.373 (3)	O5—H26	0.75 (3)
C9—H6	0.96 (2)	O5—H27	0.76 (3)
C6—C5	1.367 (3)		
N6—Ni1—N10	91.81 (5)	N11—C12—C11	122.98 (17)
N6—Ni1—N1	83.67 (5)	N11—C12—H9	117.4 (10)
N10—Ni1—N1	96.27 (5)	C11—C12—H9	119.6 (10)
N6—Ni1—N13	171.13 (5)	C10—C9—C8	119.48 (17)
N10—Ni1—N13	96.89 (5)	C10—C9—H6	122.8 (12)
N1—Ni1—N13	93.61 (5)	C8—C9—H6	117.7 (12)
N6—Ni1—N12	93.24 (5)	C5—C6—C7	119.54 (19)
N10—Ni1—N12	170.80 (5)	C5—C6—H5	121.5 (13)
N1—Ni1—N12	91.93 (5)	C7—C6—H5	118.9 (13)
N13—Ni1—N12	78.39 (5)	C11—C10—C9	119.21 (17)
N6—Ni1—N11	95.69 (5)	C11—C10—H7	120.3 (13)
N10—Ni1—N11	78.48 (5)	C9—C10—H7	120.5 (13)
N1—Ni1—N11	174.71 (5)	C5—C4—C3	118.5 (2)
N13—Ni1—N11	87.78 (5)	C5—C4—H3	121.7 (13)
N12—Ni1—N11	93.35 (5)	C3—C4—H3	119.8 (14)
N4—C1—N1	111.99 (13)	C6—C5—C4	119.27 (18)
N4—C1—N5	122.95 (13)	C6—C5—H4	119.5 (14)
N1—C1—N5	125.04 (13)	C4—C5—H4	121.2 (14)
C1—N4—N3	104.10 (12)	C22—N13—C18	118.33 (14)
C1—N1—N2	104.66 (12)	C22—N13—Ni1	126.65 (12)
C1—N1—Ni1	125.53 (10)	C18—N13—Ni1	114.95 (10)
N2—N1—Ni1	128.02 (9)	C15—C14—C13	118.61 (17)

C1—N5—C2	123.02 (13)	C15—C14—H11	121.0 (13)
C1—N5—H1	117.5 (14)	C13—C14—H11	120.4 (13)
C2—N5—H1	118.9 (14)	N13—C18—C19	121.49 (16)
C2—N6—N7	104.61 (12)	N13—C18—C17	115.73 (13)
C2—N6—Ni1	127.34 (10)	C19—C18—C17	122.78 (15)
N7—N6—Ni1	127.80 (10)	N12—C13—C14	123.10 (16)
N2—N3—N4	110.50 (12)	N12—C13—H10	117.1 (11)
N8—N7—N6	108.62 (13)	C14—C13—H10	119.8 (11)
N3—N2—N1	108.74 (12)	N12—C17—C16	121.02 (15)
N7—N8—N9	110.61 (13)	N12—C17—C18	115.58 (13)
C2—N9—N8	104.00 (13)	C16—C17—C18	123.40 (15)
N9—C2—N6	112.16 (13)	N13—C22—C21	122.71 (18)
N9—C2—N5	123.47 (14)	N13—C22—H17	117.7 (11)
N6—C2—N5	124.35 (13)	C21—C22—H17	119.6 (11)
C3—N10—C7	118.20 (14)	C15—C16—C17	119.54 (17)
C3—N10—Ni1	125.93 (11)	C15—C16—H13	124.9 (13)
C7—N10—Ni1	115.52 (11)	C17—C16—H13	115.6 (13)
C12—N11—C8	118.31 (14)	C14—C15—C16	119.40 (17)
C12—N11—Ni1	126.95 (11)	C14—C15—H12	121.7 (14)
C8—N11—Ni1	114.38 (10)	C16—C15—H12	118.9 (14)
N11—C8—C9	121.38 (16)	C20—C19—C18	119.42 (18)
N11—C8—C7	115.65 (13)	C20—C19—H14	120.2 (13)
C9—C8—C7	122.92 (15)	C18—C19—H14	120.4 (13)
N10—C7—C6	121.32 (17)	C21—C20—C19	119.28 (18)
N10—C7—C8	115.33 (13)	C21—C20—H15	120.6 (13)
C6—C7—C8	123.34 (16)	C19—C20—H15	120.1 (13)
C13—N12—C17	118.28 (13)	C20—C21—C22	118.77 (19)
C13—N12—Ni1	126.68 (11)	C20—C21—H16	121.4 (14)
C17—N12—Ni1	114.86 (10)	C22—C21—H16	119.8 (15)
C10—C11—C12	118.62 (19)	H19—O1—H18	102 (2)
C10—C11—H8	122.1 (13)	H20—O2—H21	104 (3)
C12—C11—H8	119.3 (13)	H23—O3—H22	101 (3)
N10—C3—C4	123.09 (19)	H28—O6—H29	104 (3)
N10—C3—H2	115.6 (13)	H25—O4—H24	111 (3)
C4—C3—H2	121.3 (13)	H26—O5—H27	113 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H1···O2^{i}	0.794 (19)	1.99 (2)	2.770 (2)	167.7 (19)
O1—H19···N8^{ii}	0.81 (3)	2.09 (3)	2.899 (2)	174 (3)
O1—H18···N9^{iii}	0.86 (3)	2.02 (3)	2.864 (2)	165 (3)
O2—H20···N4^{iv}	0.80 (3)	2.08 (3)	2.853 (2)	161 (3)
O2—H21···O4	0.84 (3)	1.90 (3)	2.724 (3)	168 (3)
O3—H23···N2^{v}	0.75 (3)	2.26 (3)	2.981 (2)	163 (3)
O3—H22···O1	0.82 (3)	2.08 (3)	2.885 (3)	168 (3)
O4—H25···O6^{v}	0.83 (3)	2.03 (3)	2.849 (3)	171 (3)
O4—H24···O3	0.82 (3)	2.12 (3)	2.930 (3)	167 (3)

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

Fig. 1

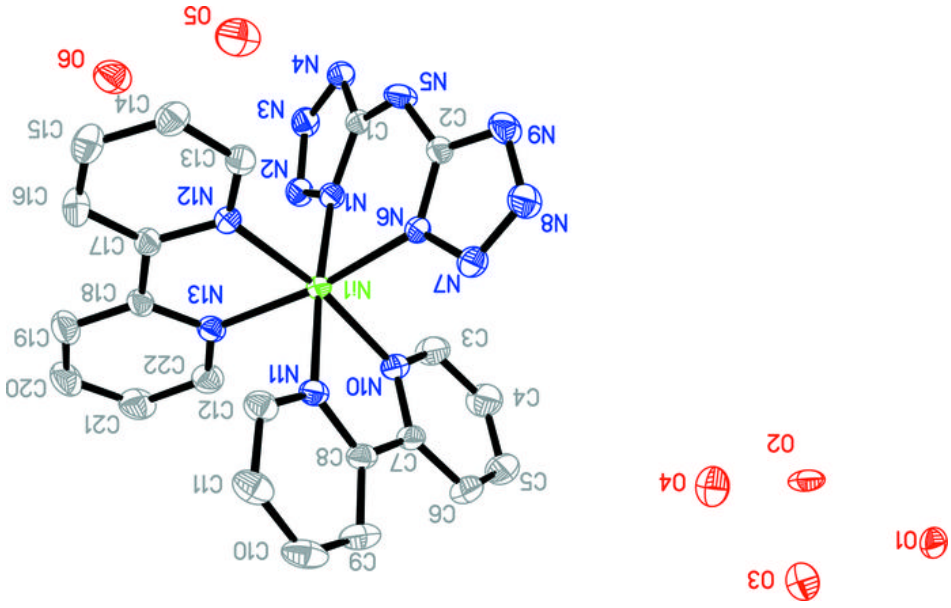

Fig. 2