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Bis[μ-4-amino-3,5-bis(hydroxymethyl)-1,2,4-triazole]bis{bis[4-amino-3,5-bis(hydroxymethyl)-1,2,4-triazole]nickel(II)} tetranitrate methanol disolvate

Yu-Peng Pan, Da-Cheng Li* and Da-Qi Wang

School of Chemistry and Chemical Engineering, Liaocheng University, Liaocheng 252059, People's Republic of China
Correspondence e-mail: dougroup@163.com

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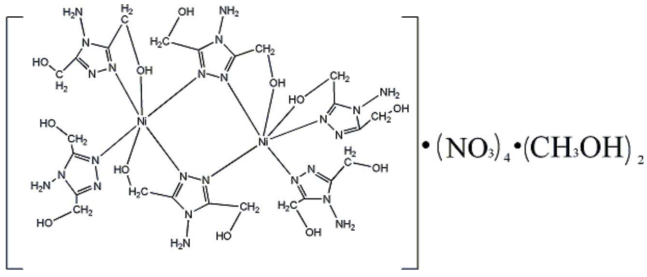
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.065; wR factor = 0.188; data-to-parameter ratio = 12.9.

The title complex, $[Ni_2(C_4H_8N_4O_2)_6](NO_3)_4\cdot 2CH_4O$, contains a centrosymmetric binuclear nickel(II) complex bridged by a pair of 4-amino-3,5-bis(hydroxymethyl)-1,2,4-triazole ligands. The separation between the Ni^{II} atoms is 3.962 (1) Å. The Ni atoms are in a slightly distorted octahedral coordination. Intermolecular N—H···O, N—H···N and O—H···O hydrogen bonds connect the ligands, solvent molecules and nitrate ions.

Related literature

For the synthesis of the ligand see: Adamek (1960). For related literature, see: Feiters (1990); Vrevgdenhil *et al.* (1987).



Experimental

Crystal data

$[Ni_2(C_4H_8N_4O_2)_6](NO_3)_4\cdot 2CH_4O$
M_r = 1294.41
Monoclinic, $P2_1/n$
a = 12.5126 (17) Å
b = 13.2808 (18) Å
c = 16.153 (2) Å
β = 92.699 (2)°
V = 2681.3 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.81 mm^{-1}
T = 298 (2) K
0.21 × 0.18 × 0.13 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.848, T_{max} = 0.902
12772 measured reflections
4719 independent reflections
2751 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.188
S = 0.91
4719 reflections
366 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −1.04 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N9	2.037 (4)	Ni1—N6^{i}	2.073 (5)
Ni1—N1	2.055 (4)	Ni1—O5	2.119 (4)
Ni1—N5	2.068 (4)	Ni1—O3	2.136 (4)
N9—Ni1—N1	104.95 (18)	N5—Ni1—O5	85.92 (15)
N9—Ni1—N5	156.10 (19)	N6^{i}—Ni1—O5	87.57 (16)
N1—Ni1—N5	92.46 (17)	N9—Ni1—O3	85.24 (16)
N9—Ni1—N6^{i}	93.72 (17)	N1—Ni1—O3	90.46 (16)
N1—Ni1—N6^{i}	91.35 (18)	N5—Ni1—O3	78.26 (16)
N5—Ni1—N6^{i}	102.25 (17)	N6^{i}—Ni1—O3	178.09 (16)
N9—Ni1—O5	77.02 (17)	O5—Ni1—O3	90.64 (15)
N1—Ni1—O5	177.81 (17)		

Symmetry code: (i) $-x+2, -y, -z+2$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4B···O8^{ii}	0.86	2.29	3.094 (8)	156
N8—H8A···O6^{iii}	0.86	2.51	3.066 (9)	123
N8—H8B···O13^{iii}	0.86	2.54	3.186 (8)	132
N12—H12B···O11^{iv}	0.86	2.25	3.027 (9)	151
O2—H2···O12^{v}	0.82	2.36	2.926 (10)	127
O2—H2···O5^{i}	0.82	2.40	3.045 (6)	136
O4—H4···O9^{vi}	0.82	1.90	2.702 (9)	166
O5—H5···N2^{i}	0.82	2.00	2.817 (6)	172
O13—H13···O1^{iii}	0.82	2.07	2.834 (7)	155

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: KP2150).

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Bis[μ-4-amino-3,5-bis(hydroxymethyl)-1,2,4-triazole]bis{bis[4-amino-3,5-bis(hydroxymethyl)-1,2,4-triazole]nickel(II)} tetranitrate methanol disolvate

Yu-Peng Pan, Da-Cheng Li and Da-Qi Wang

S1. Comment

Bridging systems based on the 1,2,4,-triazole ring are interesting due to their similarity to 1,3-imidazolate found in superoxide dismutase (Feiters, 1990). So far some polynuclear nicker(II)co-ordination compounds have been descrided with the ligand H2ahmt [4-amino-3,5-bis(hydroxymethyl)-1,2,4-triazole], such as $Ni_4(NCS)_4(Hahmt)_4(H_2O)_4$, $Ni_8(NCS)_8(ahmt)(Hahmt)_6(H_2ahmt)_4(H_2O)_{12}$ (Vrevgdenhil *et al.*, 1987). As an extension of this work on the structural characterization of 4-amino-3,5-bis(hydroxymethyl)-1,2,4-triazole derivatives, we have synthesized the title compound (I) and determined its crystal structure. The binuclear complex has a centre of symmetry and two Ni atoms are surrounded by four H_2ahmt ligands (Fig. 1, Table1). There are three distinctive coordination modes of H_2ahmt to the metal: a) the ligand acts as a tridentate ligand through the hydroxymethyl O atom and a bridging μ-N(5) andμ-N(6) coordination mode between two Ni atoms, b) as a monodentate ligand, and c) as a bidentate ligand. The finite hydrogen-bonded assembly (Fig. 2, Table 2) displays hydrogen bonds N—H···O, O—H···O and O—H···N connecting complex cations, anions, and solvent molecules.

S2. Experimental

$NiCl_2$-$6H_2O$, 4-amino-3,5-bis(hydroxymethyl)-1,2,4-triazole, and ammonia were reacted in 1:2:2 molar ratio in the mixed solution of CH_3OH and H_2O (in vol. ratio 2:1) under solvothermal condition at 423 K. After heating the solution for five days and cooling down to room temperature crystals were obtained.

S3. Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms with C—H 0.96(pyridine), C—H 0.97 (methylene) Å [U_{iso}(H) = 1.2U_{eq}(C)],*N*—H 0.86 (amino)Å [U_{iso}(H) = 1.2U_{eq}(N)], and O—H 0.82 Å (hydroxyl) [U_{iso}(H) = 1.5U_{eq}(O)]

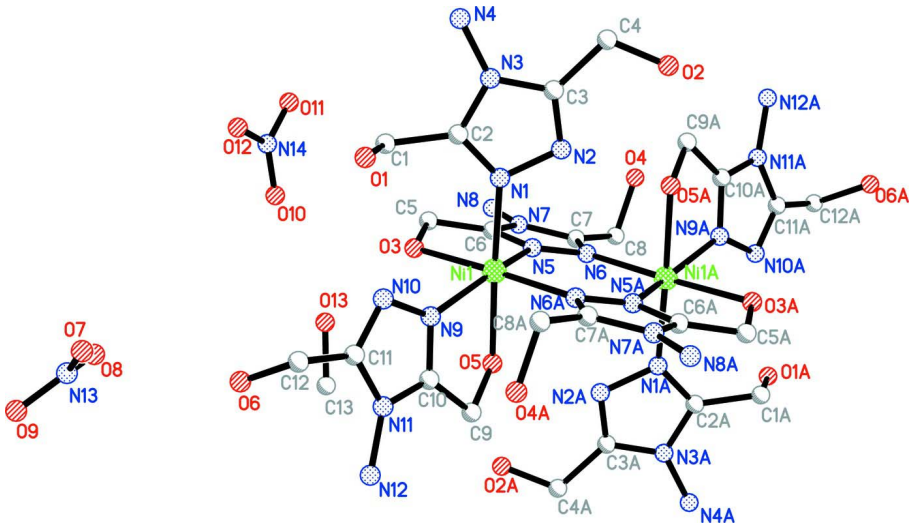

Figure 1

The molecular structure of the title complex. Displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted for clarity.

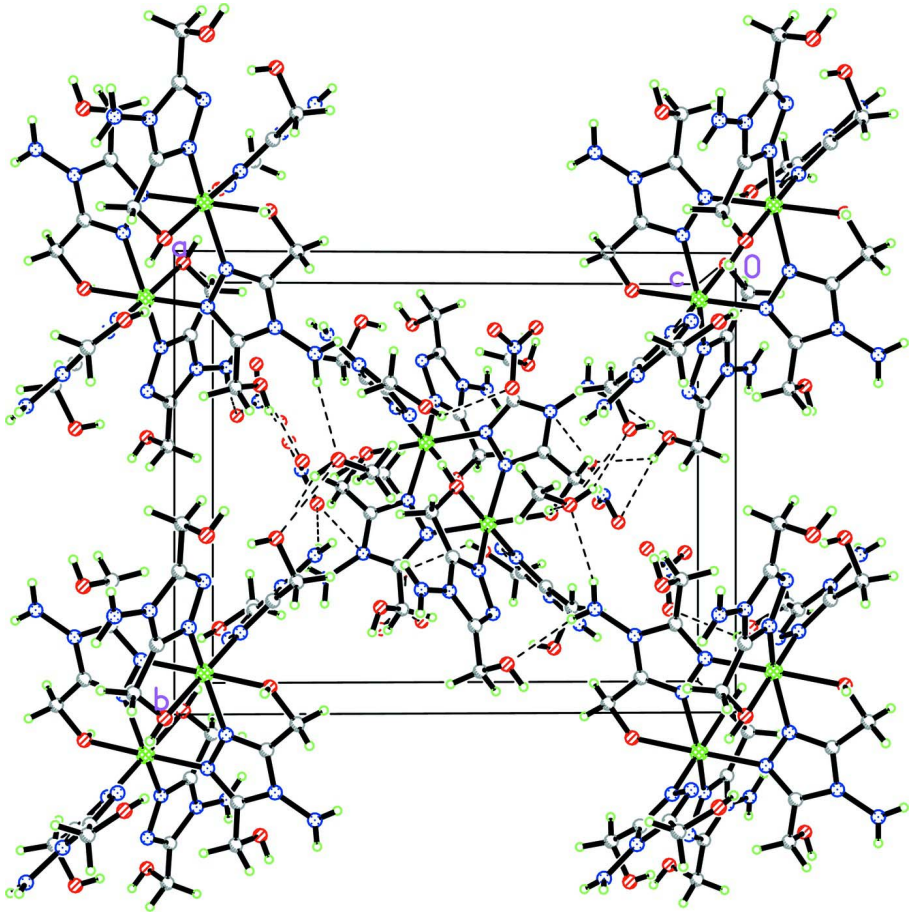

Figure 2

Crystal packing with hydrogen bonds shown as dashed lines.

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Hall symbol: -P 2yn
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$b = 13.2808\ (18)$ Å
$c = 16.153\ (2)$ Å
$\beta = 92.699\ (2)°$
$V = 2681.3\ (7)$ Å^3
$Z = 2$
$F(000) = 1344$
$D_x = 1.603$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2129 reflections
$\theta = 2.5–25.2°$
$\mu = 0.81$ mm^{-1}
$T = 298$ K
Block, green
0.21 × 0.18 × 0.13 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.848$, $T_{max} = 0.902$
12772 measured reflections
4719 independent reflections
2751 reflections with $I > 2\sigma(I)$
$R_{int} = 0.063$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$
$h = -13\rightarrow14$
$k = -15\rightarrow15$
$l = -14\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.188$
$S = 0.91$
4719 reflections
366 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.1128P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.19$ e Å^{-3}
$\Delta\rho_{min} = -1.04$ 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}
Ni1	0.90189 (5)	0.09542 (5)	0.94051 (4)	0.0325 (2)
N1	1.0196 (3)	0.1751 (3)	0.8847 (3)	0.0367 (11)
N2	1.1256 (4)	0.1555 (3)	0.9116 (3)	0.0395 (11)

N3	1.1235 (4)	0.2706 (4)	0.8165 (3)	0.0435 (12)
N4	1.1555 (4)	0.3392 (5)	0.7575 (4)	0.0728 (19)
H4A	1.1085	0.3677	0.7247	0.087*
H4B	1.2222	0.3531	0.7538	0.087*
N5	0.9656 (3)	−0.0413 (3)	0.9065 (3)	0.0337 (10)
N6	1.0247 (4)	−0.1188 (3)	0.9433 (3)	0.0358 (11)
N7	0.9660 (4)	−0.1658 (3)	0.8205 (3)	0.0421 (12)
N8	0.9454 (5)	−0.2235 (4)	0.7493 (3)	0.0707 (18)
H8A	0.9070	−0.1991	0.7085	0.085*
H8B	0.9711	−0.2833	0.7462	0.085*
N9	0.7838 (4)	0.1977 (3)	0.9584 (3)	0.0390 (11)
N10	0.7583 (4)	0.2951 (3)	0.9320 (3)	0.0411 (12)
N11	0.6360 (4)	0.2398 (4)	1.0129 (3)	0.0426 (12)
N12	0.5476 (4)	0.2333 (4)	1.0626 (3)	0.0584 (16)
H12A	0.5379	0.1800	1.0916	0.070*
H12B	0.5030	0.2824	1.0641	0.070*
N13	0.3441 (8)	0.5295 (7)	0.7903 (6)	0.1035 (16)
N14	0.7629 (7)	0.1956 (8)	0.6076 (6)	0.098 (3)
O1	0.8962 (4)	0.3815 (3)	0.8150 (3)	0.0641 (13)
H1	0.8593	0.3713	0.8547	0.096*
O2	1.3471 (4)	0.1683 (4)	0.9440 (3)	0.0683 (14)
H2	1.2991	0.1490	0.9730	0.103*
O3	0.8214 (3)	0.0701 (3)	0.8228 (2)	0.0417 (10)
H3	0.7570	0.0621	0.8274	0.062*
O4	1.1786 (4)	−0.2835 (4)	0.8582 (3)	0.0828 (17)
H4	1.1977	−0.3393	0.8430	0.124*
O5	0.7851 (3)	0.0094 (3)	0.9998 (2)	0.0406 (9)
H5	0.8048	−0.0402	1.0267	0.061*
O6	0.5163 (5)	0.3947 (4)	0.9096 (4)	0.1035 (16)
H6	0.5003	0.4432	0.8801	0.155*
O7	0.4045 (6)	0.5941 (5)	0.8164 (5)	0.125 (3)
O8	0.3724 (5)	0.4514 (5)	0.7620 (5)	0.115 (3)
O9	0.2502 (7)	0.5485 (6)	0.7845 (6)	0.159 (4)
O10	0.7076 (6)	0.1478 (7)	0.6485 (5)	0.144 (3)
O11	0.8346 (7)	0.1599 (6)	0.5698 (6)	0.157 (4)
O12	0.7564 (6)	0.2863 (8)	0.6035 (5)	0.134 (3)
O13	0.6140 (4)	0.0487 (4)	0.7942 (4)	0.0797 (16)
H13	0.6044	0.0139	0.7526	0.120*
C1	0.9259 (5)	0.2882 (5)	0.7800 (4)	0.0494 (16)
H1A	0.9426	0.2981	0.7226	0.059*
H1B	0.8663	0.2415	0.7813	0.059*
C2	1.0214 (5)	0.2441 (4)	0.8270 (4)	0.0400 (14)
C3	1.1843 (4)	0.2153 (4)	0.8701 (4)	0.0389 (13)
C4	1.3038 (5)	0.2228 (5)	0.8783 (4)	0.0545 (17)
H4C	1.3336	0.1988	0.8276	0.065*
H4D	1.3238	0.2929	0.8853	0.065*
C5	0.8627 (5)	−0.0106 (5)	0.7768 (4)	0.0461 (15)
H5A	0.9039	0.0151	0.7320	0.055*

H5B	0.8046	−0.0513	0.7530	0.055*
C6	0.9310 (5)	−0.0715 (4)	0.8334 (3)	0.0372 (13)
C7	1.0245 (5)	−0.1936 (4)	0.8897 (4)	0.0407 (14)
C8	1.0773 (6)	−0.2919 (4)	0.8967 (4)	0.0530 (17)
H8C	1.0883	−0.3106	0.9545	0.064*
H8D	1.0334	−0.3431	0.8690	0.064*
C9	0.7120 (5)	0.0662 (4)	1.0460 (4)	0.0446 (15)
H9A	0.7365	0.0708	1.1037	0.054*
H9B	0.6415	0.0356	1.0427	0.054*
C10	0.7096 (4)	0.1669 (4)	1.0066 (3)	0.0367 (13)
C11	0.6685 (5)	0.3183 (4)	0.9657 (4)	0.0410 (14)
C12	0.6129 (6)	0.4159 (5)	0.9566 (5)	0.063 (2)
H12C	0.5976	0.4433	1.0104	0.076*
H12D	0.6562	0.4638	0.9277	0.076*
C13	0.5390 (7)	0.0235 (7)	0.8515 (6)	0.093 (3)
H13A	0.5649	−0.0324	0.8843	0.140*
H13B	0.4728	0.0054	0.8228	0.140*
H13C	0.5275	0.0801	0.8869	0.140*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0297 (4)	0.0320 (4)	0.0364 (4)	0.0031 (3)	0.0070 (3)	0.0008 (3)
N1	0.030 (3)	0.037 (3)	0.044 (3)	0.002 (2)	0.008 (2)	0.005 (2)
N2	0.031 (3)	0.042 (3)	0.046 (3)	0.002 (2)	0.008 (2)	0.008 (2)
N3	0.039 (3)	0.045 (3)	0.047 (3)	−0.006 (2)	0.008 (2)	0.012 (2)
N4	0.044 (3)	0.089 (5)	0.086 (4)	−0.015 (3)	0.001 (3)	0.052 (4)
N5	0.031 (3)	0.034 (2)	0.037 (3)	0.004 (2)	0.005 (2)	0.000 (2)
N6	0.038 (3)	0.029 (2)	0.042 (3)	0.007 (2)	0.012 (2)	−0.002 (2)
N7	0.049 (3)	0.039 (3)	0.039 (3)	0.000 (2)	0.003 (2)	−0.009 (2)
N8	0.104 (5)	0.057 (4)	0.049 (4)	0.009 (4)	−0.016 (3)	−0.020 (3)
N9	0.037 (3)	0.037 (3)	0.043 (3)	0.009 (2)	0.008 (2)	0.003 (2)
N10	0.041 (3)	0.037 (3)	0.046 (3)	0.010 (2)	0.007 (2)	0.002 (2)
N11	0.037 (3)	0.045 (3)	0.046 (3)	0.009 (2)	0.007 (2)	−0.003 (2)
N12	0.042 (3)	0.066 (4)	0.070 (4)	0.022 (3)	0.030 (3)	0.018 (3)
N13	0.081 (4)	0.088 (3)	0.141 (5)	0.008 (3)	−0.005 (3)	−0.001 (3)
N14	0.071 (6)	0.107 (7)	0.117 (7)	0.008 (6)	0.013 (5)	0.029 (6)
O1	0.067 (3)	0.056 (3)	0.071 (3)	0.015 (2)	0.021 (3)	0.020 (2)
O2	0.042 (3)	0.073 (3)	0.089 (4)	−0.009 (2)	0.002 (3)	0.023 (3)
O3	0.037 (2)	0.042 (2)	0.046 (2)	0.0036 (18)	0.0040 (19)	0.0000 (18)
O4	0.088 (4)	0.062 (3)	0.100 (4)	0.029 (3)	0.026 (3)	−0.010 (3)
O5	0.040 (2)	0.035 (2)	0.049 (2)	0.0061 (18)	0.0154 (18)	−0.0010 (17)
O6	0.081 (4)	0.088 (3)	0.141 (5)	0.008 (3)	−0.005 (3)	−0.001 (3)
O7	0.115 (6)	0.117 (5)	0.145 (6)	−0.035 (5)	0.044 (5)	−0.058 (5)
O8	0.085 (4)	0.067 (4)	0.198 (7)	−0.004 (3)	0.057 (5)	−0.030 (4)
O9	0.106 (6)	0.126 (6)	0.242 (10)	0.038 (5)	−0.014 (6)	−0.079 (6)
O10	0.108 (6)	0.181 (8)	0.147 (7)	−0.009 (6)	0.051 (5)	0.045 (6)
O11	0.118 (6)	0.113 (6)	0.248 (10)	−0.012 (5)	0.098 (7)	−0.013 (6)

O12	0.115 (6)	0.147 (7)	0.142 (7)	0.037 (6)	0.014 (5)	0.024 (6)
O13	0.061 (3)	0.083 (4)	0.097 (4)	−0.012 (3)	0.023 (3)	−0.016 (3)
C1	0.048 (4)	0.050 (4)	0.050 (4)	0.004 (3)	0.006 (3)	0.012 (3)
C2	0.040 (4)	0.038 (3)	0.042 (3)	−0.004 (3)	0.004 (3)	0.004 (3)
C3	0.032 (3)	0.042 (3)	0.044 (3)	−0.005 (3)	0.009 (3)	0.005 (3)
C4	0.042 (4)	0.057 (4)	0.065 (5)	−0.013 (3)	0.002 (3)	0.013 (3)
C5	0.046 (4)	0.053 (4)	0.039 (3)	0.012 (3)	0.002 (3)	−0.007 (3)
C6	0.038 (3)	0.039 (3)	0.035 (3)	0.003 (3)	0.004 (3)	−0.002 (2)
C7	0.043 (4)	0.037 (3)	0.042 (4)	0.003 (3)	0.009 (3)	−0.003 (3)
C8	0.067 (5)	0.037 (3)	0.054 (4)	0.005 (3)	−0.003 (3)	0.000 (3)
C9	0.041 (4)	0.045 (3)	0.049 (4)	0.007 (3)	0.016 (3)	0.002 (3)
C10	0.029 (3)	0.038 (3)	0.043 (3)	0.004 (3)	0.006 (3)	−0.001 (3)
C11	0.038 (3)	0.042 (3)	0.043 (3)	0.007 (3)	0.004 (3)	0.001 (3)
C12	0.066 (5)	0.052 (4)	0.074 (5)	0.027 (4)	0.012 (4)	0.007 (4)
C13	0.082 (6)	0.102 (7)	0.100 (7)	−0.041 (5)	0.035 (5)	−0.024 (5)

Geometric parameters (Å, º)

Ni1—N9	2.037 (4)	N14—O12	1.209 (10)
Ni1—N1	2.055 (4)	O1—C1	1.419 (7)
Ni1—N5	2.068 (4)	O1—H1	0.8200
Ni1—N6^{i}	2.073 (5)	O2—C4	1.374 (7)
Ni1—O5	2.119 (4)	O2—H2	0.8200
Ni1—O3	2.136 (4)	O3—C5	1.415 (6)
N1—C2	1.308 (7)	O3—H3	0.8200
N1—N2	1.402 (6)	O4—C8	1.443 (8)
N2—C3	1.291 (7)	O4—H4	0.8200
N3—C2	1.343 (7)	O5—C9	1.424 (6)
N3—C3	1.344 (7)	O5—H5	0.8200
N3—N4	1.392 (6)	O6—C12	1.424 (9)
N4—H4A	0.8600	O6—H6	0.8200
N4—H4B	0.8600	O13—C13	1.389 (8)
N5—C6	1.302 (7)	O13—H13	0.8200
N5—N6	1.385 (6)	C1—C2	1.505 (8)
N6—C7	1.318 (7)	C1—H1A	0.9700
N6—Ni1^{i}	2.073 (5)	C1—H1B	0.9700
N7—C6	1.347 (7)	C3—C4	1.498 (8)
N7—C7	1.359 (7)	C4—H4C	0.9700
N7—N8	1.395 (6)	C4—H4D	0.9700
N8—H8A	0.8600	C5—C6	1.465 (8)
N8—H8B	0.8600	C5—H5A	0.9700
N9—C10	1.305 (7)	C5—H5B	0.9700
N9—N10	1.395 (6)	C7—C8	1.465 (8)
N10—C11	1.308 (7)	C8—H8C	0.9700
N11—C10	1.343 (7)	C8—H8D	0.9700
N11—C11	1.363 (7)	C9—C10	1.481 (8)
N11—N12	1.400 (6)	C9—H9A	0.9700
N12—H12A	0.8600	C9—H9B	0.9700

N12—H12B	0.8600	C11—C12	1.475 (8)
N13—O8	1.194 (9)	C12—H12C	0.9700
N13—O9	1.202 (10)	C12—H12D	0.9700
N13—O7	1.207 (10)	C13—H13A	0.9600
N14—O10	1.167 (9)	C13—H13B	0.9600
N14—O11	1.205 (10)	C13—H13C	0.9600

N9—Ni1—N1	104.95 (18)	C12—O6—H6	109.5
N9—Ni1—N5	156.10 (19)	C13—O13—H13	109.5
N1—Ni1—N5	92.46 (17)	O1—C1—C2	110.8 (5)
N9—Ni1—N6^{i}	93.72 (17)	O1—C1—H1A	109.5
N1—Ni1—N6^{i}	91.35 (18)	C2—C1—H1A	109.5
N5—Ni1—N6^{i}	102.25 (17)	O1—C1—H1B	109.5
N9—Ni1—O5	77.02 (17)	C2—C1—H1B	109.5
N1—Ni1—O5	177.81 (17)	H1A—C1—H1B	108.1
N5—Ni1—O5	85.92 (15)	N1—C2—N3	108.8 (5)
N6^{i}—Ni1—O5	87.57 (16)	N1—C2—C1	126.3 (5)
N9—Ni1—O3	85.24 (16)	N3—C2—C1	124.9 (5)
N1—Ni1—O3	90.46 (16)	N2—C3—N3	110.7 (5)
N5—Ni1—O3	78.26 (16)	N2—C3—C4	125.8 (5)
N6^{i}—Ni1—O3	178.09 (16)	N3—C3—C4	123.6 (5)
O5—Ni1—O3	90.64 (15)	O2—C4—C3	112.9 (5)
C2—N1—N2	107.6 (4)	O2—C4—H4C	109.0
C2—N1—Ni1	135.3 (4)	C3—C4—H4C	109.0
N2—N1—Ni1	117.1 (3)	O2—C4—H4D	109.0
C3—N2—N1	106.1 (4)	C3—C4—H4D	109.0
C2—N3—C3	106.8 (5)	H4C—C4—H4D	107.8
C2—N3—N4	124.3 (5)	O3—C5—C6	107.9 (5)
C3—N3—N4	128.8 (5)	O3—C5—H5A	110.1
N3—N4—H4A	120.0	C6—C5—H5A	110.1
N3—N4—H4B	120.0	O3—C5—H5B	110.1
H4A—N4—H4B	120.0	C6—C5—H5B	110.1
C6—N5—N6	108.0 (4)	H5A—C5—H5B	108.4
C6—N5—Ni1	113.3 (4)	N5—C6—N7	109.3 (5)
N6—N5—Ni1	138.0 (3)	N5—C6—C5	123.8 (5)
C7—N6—N5	107.1 (5)	N7—C6—C5	127.0 (5)
C7—N6—Ni1^{i}	134.0 (4)	N6—C7—N7	108.7 (5)
N5—N6—Ni1^{i}	118.9 (3)	N6—C7—C8	129.4 (6)
C6—N7—C7	107.0 (5)	N7—C7—C8	121.9 (5)
C6—N7—N8	126.1 (5)	O4—C8—C7	107.5 (5)
C7—N7—N8	126.9 (5)	O4—C8—H8C	110.2
N7—N8—H8A	120.0	C7—C8—H8C	110.2
N7—N8—H8B	120.0	O4—C8—H8D	110.2
H8A—N8—H8B	120.0	C7—C8—H8D	110.2
C10—N9—N10	108.3 (4)	H8C—C8—H8D	108.5
C10—N9—Ni1	114.6 (4)	O5—C9—C10	104.7 (4)
N10—N9—Ni1	137.1 (4)	O5—C9—H9A	110.8
C11—N10—N9	106.2 (5)	C10—C9—H9A	110.8

C10—N11—C11	106.6 (5)	O5—C9—H9B	110.8
C10—N11—N12	124.1 (5)	C10—C9—H9B	110.8
C11—N11—N12	129.2 (5)	H9A—C9—H9B	108.9
N11—N12—H12A	120.0	N9—C10—N11	109.2 (5)
N11—N12—H12B	120.0	N9—C10—C9	122.6 (5)
H12A—N12—H12B	120.0	N11—C10—C9	128.3 (5)
O8—N13—O9	117.3 (10)	N10—C11—N11	109.7 (5)
O8—N13—O7	124.0 (10)	N10—C11—C12	125.1 (6)
O9—N13—O7	118.2 (10)	N11—C11—C12	125.1 (5)
O10—N14—O11	123.3 (11)	O6—C12—C11	105.4 (6)
O10—N14—O12	122.2 (11)	O6—C12—H12C	110.7
O11—N14—O12	114.5 (10)	C11—C12—H12C	110.7
C1—O1—H1	109.5	O6—C12—H12D	110.7
C4—O2—H2	109.5	C11—C12—H12D	110.7
C5—O3—Ni1	114.7 (3)	H12C—C12—H12D	108.8
C5—O3—H3	109.5	O13—C13—H13A	109.5
Ni1—O3—H3	111.3	O13—C13—H13B	109.5
C8—O4—H4	109.5	H13A—C13—H13B	109.5
C9—O5—Ni1	115.1 (3)	O13—C13—H13C	109.5
C9—O5—H5	109.4	H13A—C13—H13C	109.5
Ni1—O5—H5	118.4	H13B—C13—H13C	109.5

Symmetry code: (i) $-x+2, -y, -z+2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*B*···O8^{ii}	0.86	2.29	3.094 (8)	156
N8—H8*A*···O6^{iii}	0.86	2.51	3.066 (9)	123
N8—H8*B*···O13^{iii}	0.86	2.54	3.186 (8)	132
N12—H12*B*···O11^{iv}	0.86	2.25	3.027 (9)	151
O2—H2···O12^{v}	0.82	2.36	2.926 (10)	127
O2—H2···O5^{i}	0.82	2.40	3.045 (6)	136
O4—H4···O9^{vi}	0.82	1.90	2.702 (9)	166
O5—H5···N2^{i}	0.82	2.00	2.817 (6)	172
O13—H13···O1^{iii}	0.82	2.07	2.834 (7)	155

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