Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ammonium tris(3-aminopyrazine-2-carboxylato-κ^2N^1,O)nickelate(II) trihydrate

Xiao-Li Cheng,[a] Shan Gao[a] and Seik Weng Ng[b]*

[a]College of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

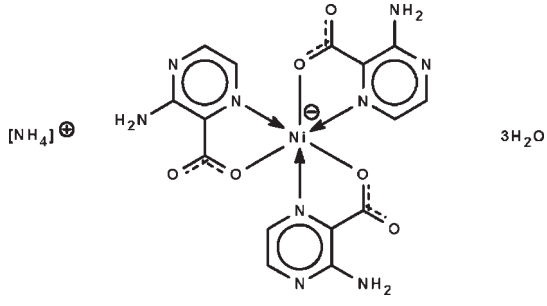
Received 14 November 2009; accepted 14 November 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.096; data-to-parameter ratio = 13.5.

The Ni^{II} atom in the title hydrated salt, $(NH_4)[Ni(C_5H_4N_3O_2)_3]\cdot 3H_2O$, is N,O-chelated by the three 3-aminopyrazine-2-carboxylate ligands, resulting in a distorted octahedral *mer*-NiN_3O_3 geometry for the metal. In the crystal, the complex anion, ammonium cation and uncoordinated water molecules are linked by extensive N—H···N, N—H···O, O—H···N and O—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

For the crystal structure of diaquabis(3-aminopyrazine-2-carboxylato)nickel(II), see: Ptasiewicz-Bak & Leciejewicz (1999).



Experimental

Crystal data

$(NH_4)[Ni(C_5H_4N_3O_2)_3]\cdot 3H_2O$
M_r = 545.14
Monoclinic, $P2_1/n$
a = 11.2092 (3) Å
b = 14.7061 (4) Å
c = 13.7540 (4) Å
β = 97.5214 (8)°
V = 2247.75 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.93 mm^{-1}
T = 293 K
0.28 × 0.22 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.780, T_{max} = 0.843
21341 measured reflections
5114 independent reflections
4322 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.096
S = 1.02
5114 reflections
380 parameters
25 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O1	2.0476 (13)	Ni1—N7	2.0561 (14)
Ni1—O3	2.0526 (13)	Ni1—N4	2.0805 (14)
Ni1—O5	2.0567 (13)	Ni1—N1	2.0857 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H31···O2	0.85 (1)	2.08 (2)	2.740 (3)	134 (2)
N6—H61···O4	0.85 (1)	2.01 (2)	2.701 (3)	138 (2)
N6—H62···N5^{i}	0.85 (1)	2.15 (1)	2.992 (2)	175 (2)
N9—H91···O6	0.86 (1)	2.07 (2)	2.733 (2)	134 (2)
N9—H92···O3^{ii}	0.85 (1)	2.10 (1)	2.924 (2)	164 (2)
N10—H101···O2	0.86 (1)	1.91 (1)	2.756 (3)	169 (3)
N10—H102···O1w	0.86 (1)	1.94 (1)	2.779 (3)	164 (3)
N10—H103···O2w	0.85 (1)	2.07 (1)	2.919 (3)	172 (3)
N10—H104···N8^{iii}	0.84 (1)	2.36 (2)	3.018 (3)	135 (2)
O1w—H1w1···O1	0.84 (1)	2.25 (2)	2.964 (3)	143 (4)
O1w—H1w2···N2^{iv}	0.85 (1)	2.00 (1)	2.842 (3)	171 (4)
O2w—H2w1···O5^{v}	0.85 (1)	2.03 (1)	2.869 (2)	168 (3)
O2w—H2w2···O6^{vi}	0.84 (1)	1.96 (1)	2.766 (2)	159 (3)
O3w—H3w1···O4	0.85 (1)	2.39 (5)	2.812 (3)	111 (4)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the Key Project of the Natural Science Foundation of Heilongjiang Province (No. ZD200903), the Scientific Fund of Remarkable Teachers of Heilongjiang Province (No. 1054 G036), Heilongjiang University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5228).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Ptasiewicz-Bak, H. & Leciejewicz, B. (1999). *Pol. J. Chem.* **73**, 717–725.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalClear*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2009). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2009). E**65**, m1631-m1632 [**doi:10.1107/S1600536809048363**]

Ammonium tris(3-aminopyrazine-2-carboxylato-κ^2N^1,O)nickelate(II) trihydrate

X.-L. Cheng, S. Gao and S. W. Ng

Experimental

Nickel dichloride hexahydrate (0.48 g, 2 mmol), 3-aminopyrazine-2-carboxylic acid (0.56 g, 4 mmol) and sodium hydroxide (0.16 g 4 mmol) were dissolved in a water/DMF (*v*/*v* = 10 ml:1 ml) mixture. The solution was sealed in a 50 ml Teflon-lined stainless steel bomb and held at 443 K for 3 days. The bomb was gradually cooled to room temperature;, and green blocks of (I) were obtained after several days. The presence of the ammonium counterion is explained by the decomposition of DMF.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93 Å) and were included in the refinement in the riding model approximation, with *U*(H) = 1.2*U*(C). The amino and water H-atoms were located in a difference Fourier map, and were refined with a distance restraint of N–H = O–H = 0.85±0.01 Å; their U_{iso} values were refined.

Figures

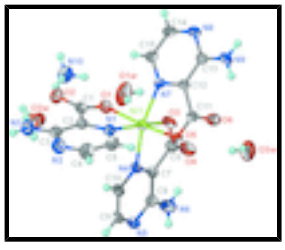


Fig. 1. The molecular structure of (I) at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Ammonium tris(3-aminopyrazine-2-carboxylato-κ^2N^1,O)nickelate(II) trihydrate

Crystal data

$(NH_4)[Ni(C_5H_4N_3O_2)_3]\cdot 3H_2O$	F_{000} = 1128
M_r = 545.14	D_x = 1.611 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 17239 reflections
a = 11.2092 (3) Å	θ = 3.2–27.5º
b = 14.7061 (4) Å	μ = 0.93 mm^{-1}
c = 13.7540 (4) Å	*T* = 293 K
β = 97.5214 (8)º	Block, green
V = 2247.75 (11) Å^3	0.28 × 0.22 × 0.19 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID IP diffractometer	5114 independent reflections
Radiation source: fine-focus sealed tube	4322 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.028$
$T = 293$ K	$\theta_{max} = 27.4^{o}$
ω scan	$\theta_{min} = 3.2^{o}$
Absorption correction: Multi-scan (ABSCOR; Higashi, 1995)	$h = -14 \rightarrow 14$
$T_{min} = 0.780$, $T_{max} = 0.843$	$k = -19 \rightarrow 19$
21341 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.096$	$w = 1/[\sigma^2(F_o^2) + (0.0582P)^2 + 0.5222P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
5114 reflections	$\Delta\rho_{max} = 0.47$ e Å^{-3}
380 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
25 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.31748 (2)	0.668273 (15)	0.689311 (16)	0.03143 (9)
O1	0.42705 (12)	0.74401 (9)	0.78914 (10)	0.0399 (3)
O2	0.46912 (13)	0.77268 (10)	0.94948 (10)	0.0483 (3)
O3	0.37891 (13)	0.72843 (9)	0.57068 (10)	0.0446 (3)
O4	0.33775 (17)	0.83556 (12)	0.45711 (12)	0.0616 (5)
O5	0.21564 (11)	0.57245 (9)	0.60709 (9)	0.0391 (3)
O6	0.22619 (12)	0.43404 (11)	0.54208 (12)	0.0536 (4)
O1W	0.5686 (2)	0.8674 (2)	0.67787 (15)	0.0930 (8)
O2W	0.53958 (16)	1.06663 (15)	0.88906 (13)	0.0643 (5)
O3W	0.3437 (3)	0.8148 (3)	0.25454 (17)	0.0980 (8)
N1	0.25466 (14)	0.62854 (10)	0.81904 (11)	0.0341 (3)
N2	0.15754 (17)	0.62107 (13)	0.99403 (13)	0.0500 (4)
N3	0.2905 (2)	0.73064 (15)	1.06019 (13)	0.0525 (5)
N4	0.19233 (13)	0.77229 (10)	0.65903 (10)	0.0327 (3)
N5	0.03798 (15)	0.91375 (11)	0.59648 (12)	0.0410 (4)

N6	0.14458 (19)	0.94291 (14)	0.46855 (15)	0.0560 (5)
N7	0.44529 (13)	0.56953 (10)	0.67977 (10)	0.0307 (3)
N8	0.60635 (14)	0.44427 (11)	0.62184 (12)	0.0389 (4)
N9	0.45018 (16)	0.36067 (12)	0.54171 (13)	0.0418 (4)
N10	0.6367 (2)	0.88322 (17)	0.87878 (16)	0.0609 (5)
C1	0.40689 (16)	0.73693 (12)	0.87734 (13)	0.0352 (4)
C2	0.30163 (16)	0.67882 (12)	0.89515 (13)	0.0331 (4)
C3	0.25094 (18)	0.67630 (13)	0.98471 (14)	0.0389 (4)
C4	0.1158 (2)	0.56992 (16)	0.91715 (16)	0.0528 (5)
H4	0.0521	0.5307	0.9229	0.063*
C5	0.16254 (19)	0.57242 (14)	0.82959 (15)	0.0429 (4)
H5	0.1305	0.5354	0.7778	0.052*
C6	0.31612 (18)	0.79401 (13)	0.53101 (13)	0.0385 (4)
C7	0.21035 (17)	0.82128 (11)	0.58056 (13)	0.0321 (4)
C8	0.13109 (17)	0.89357 (12)	0.54759 (13)	0.0368 (4)
C9	0.02365 (18)	0.86309 (14)	0.67384 (14)	0.0413 (4)
H9	−0.0410	0.8758	0.7077	0.050*
C10	0.09911 (17)	0.79277 (14)	0.70670 (13)	0.0390 (4)
H10	0.0855	0.7596	0.7618	0.047*
C11	0.27182 (16)	0.50055 (13)	0.58777 (13)	0.0345 (4)
C12	0.40521 (15)	0.49993 (12)	0.62341 (12)	0.0294 (3)
C13	0.48649 (16)	0.43358 (12)	0.59462 (12)	0.0316 (4)
C14	0.64164 (17)	0.51541 (14)	0.67792 (15)	0.0418 (4)
H14	0.7237	0.5235	0.6970	0.050*
C15	0.56347 (16)	0.57786 (13)	0.70937 (14)	0.0379 (4)
H15	0.5924	0.6252	0.7507	0.045*
H1W1	0.518 (4)	0.826 (3)	0.681 (3)	0.16 (2)*
H1W2	0.587 (4)	0.871 (3)	0.6200 (14)	0.138 (16)*
H2W1	0.4664 (10)	1.071 (2)	0.8988 (19)	0.079 (10)*
H2W2	0.586 (2)	1.078 (3)	0.9408 (15)	0.123 (15)*
H3W1	0.366 (6)	0.861 (2)	0.290 (3)	0.20 (3)*
H3W2	0.325 (6)	0.772 (3)	0.292 (3)	0.21 (3)*
H31	0.3539 (16)	0.7617 (16)	1.056 (2)	0.064 (8)*
H32	0.259 (2)	0.724 (2)	1.1133 (13)	0.076 (9)*
H61	0.2078 (15)	0.9305 (16)	0.4426 (16)	0.054 (7)*
H62	0.0924 (18)	0.9821 (13)	0.4466 (18)	0.058 (7)*
H91	0.3745 (10)	0.3567 (18)	0.5212 (19)	0.059 (7)*
H92	0.5008 (17)	0.3270 (13)	0.5183 (17)	0.048 (7)*
H101	0.592 (2)	0.8458 (15)	0.9058 (17)	0.079 (10)*
H102	0.627 (3)	0.871 (2)	0.8169 (8)	0.106 (13)*
H103	0.612 (3)	0.9371 (9)	0.887 (2)	0.16 (2)*
H104	0.7095 (10)	0.876 (2)	0.902 (2)	0.124 (15)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.03054 (14)	0.03344 (14)	0.03110 (13)	0.00615 (9)	0.00699 (9)	0.00153 (9)
O1	0.0392 (7)	0.0421 (7)	0.0402 (7)	−0.0052 (6)	0.0123 (6)	−0.0010 (6)

O2	0.0463 (8)	0.0536 (8)	0.0440 (7)	−0.0107 (7)	0.0029 (6)	−0.0063 (7)
O3	0.0452 (8)	0.0472 (7)	0.0454 (7)	0.0188 (6)	0.0214 (6)	0.0114 (6)
O4	0.0649 (11)	0.0760 (11)	0.0496 (9)	0.0243 (9)	0.0285 (8)	0.0262 (8)
O5	0.0292 (6)	0.0472 (7)	0.0400 (7)	0.0099 (6)	0.0012 (5)	−0.0059 (6)
O6	0.0326 (7)	0.0540 (9)	0.0713 (10)	0.0016 (6)	−0.0046 (7)	−0.0237 (8)
O1W	0.0979 (17)	0.131 (2)	0.0520 (11)	−0.0539 (16)	0.0174 (11)	−0.0013 (12)
O2W	0.0414 (9)	0.0925 (13)	0.0577 (10)	−0.0038 (9)	0.0021 (8)	−0.0103 (10)
O3W	0.0917 (18)	0.144 (2)	0.0590 (12)	−0.0021 (17)	0.0121 (12)	−0.0216 (15)
N1	0.0345 (8)	0.0350 (8)	0.0325 (7)	0.0019 (6)	0.0037 (6)	0.0027 (6)
N2	0.0528 (11)	0.0581 (11)	0.0419 (9)	−0.0058 (9)	0.0163 (8)	0.0059 (8)
N3	0.0585 (12)	0.0643 (12)	0.0360 (9)	−0.0039 (10)	0.0113 (9)	−0.0049 (9)
N4	0.0335 (8)	0.0342 (7)	0.0312 (7)	0.0067 (6)	0.0067 (6)	−0.0003 (6)
N5	0.0408 (9)	0.0391 (8)	0.0427 (8)	0.0131 (7)	0.0042 (7)	−0.0013 (7)
N6	0.0556 (12)	0.0564 (11)	0.0582 (11)	0.0224 (10)	0.0161 (10)	0.0245 (10)
N7	0.0276 (7)	0.0340 (7)	0.0303 (7)	0.0042 (6)	0.0032 (6)	0.0011 (6)
N8	0.0298 (8)	0.0432 (8)	0.0443 (8)	0.0071 (7)	0.0074 (7)	0.0015 (7)
N9	0.0349 (9)	0.0419 (9)	0.0498 (9)	0.0042 (7)	0.0107 (8)	−0.0092 (8)
N10	0.0507 (12)	0.0779 (16)	0.0551 (12)	−0.0199 (11)	0.0108 (10)	−0.0007 (11)
C1	0.0343 (9)	0.0340 (9)	0.0374 (9)	0.0032 (7)	0.0053 (7)	−0.0006 (8)
C2	0.0325 (9)	0.0349 (9)	0.0317 (8)	0.0058 (7)	0.0037 (7)	0.0029 (7)
C3	0.0408 (10)	0.0432 (10)	0.0329 (9)	0.0067 (8)	0.0054 (8)	0.0042 (8)
C4	0.0518 (13)	0.0584 (13)	0.0501 (12)	−0.0145 (11)	0.0138 (10)	0.0067 (11)
C5	0.0442 (11)	0.0436 (10)	0.0407 (10)	−0.0084 (9)	0.0046 (8)	0.0021 (8)
C6	0.0408 (10)	0.0426 (10)	0.0335 (9)	0.0070 (8)	0.0104 (8)	0.0023 (8)
C7	0.0338 (9)	0.0319 (8)	0.0306 (8)	0.0043 (7)	0.0039 (7)	−0.0011 (7)
C8	0.0387 (10)	0.0340 (9)	0.0368 (9)	0.0043 (8)	0.0014 (8)	0.0007 (7)
C9	0.0376 (10)	0.0478 (11)	0.0393 (9)	0.0113 (9)	0.0084 (8)	−0.0069 (9)
C10	0.0387 (10)	0.0461 (10)	0.0339 (9)	0.0089 (8)	0.0111 (8)	−0.0001 (8)
C11	0.0280 (8)	0.0433 (10)	0.0321 (8)	0.0044 (7)	0.0036 (7)	−0.0003 (8)
C12	0.0269 (8)	0.0330 (8)	0.0288 (8)	0.0038 (7)	0.0050 (6)	0.0027 (7)
C13	0.0315 (9)	0.0335 (8)	0.0306 (8)	0.0042 (7)	0.0076 (7)	0.0037 (7)
C14	0.0253 (9)	0.0500 (11)	0.0496 (11)	0.0033 (8)	0.0027 (8)	−0.0003 (9)
C15	0.0304 (9)	0.0412 (10)	0.0409 (9)	0.0008 (8)	0.0006 (7)	−0.0029 (8)

Geometric parameters (Å, °)

Ni1—O1	2.0476 (13)	N6—C8	1.332 (3)
Ni1—O3	2.0526 (13)	N6—H61	0.853 (10)
Ni1—O5	2.0567 (13)	N6—H62	0.849 (10)
Ni1—N7	2.0561 (14)	N7—C12	1.326 (2)
Ni1—N4	2.0805 (14)	N7—C15	1.340 (2)
Ni1—N1	2.0857 (15)	N8—C14	1.329 (3)
O1—C1	1.267 (2)	N8—C13	1.356 (2)
O2—C1	1.252 (2)	N9—C13	1.329 (2)
O3—C6	1.274 (2)	N9—H91	0.860 (10)
O4—C6	1.237 (2)	N9—H92	0.848 (10)
O5—C11	1.276 (2)	N10—H101	0.861 (9)
O6—C11	1.237 (2)	N10—H102	0.861 (9)
O1W—H1W1	0.84 (1)	N10—H103	0.853 (9)

O1W—H1W2	0.85 (1)	N10—H104	0.844 (9)
O2W—H2W1	0.85 (1)	C1—C2	1.503 (3)
O2W—H2W2	0.84 (1)	C2—C3	1.423 (3)
O3W—H3W1	0.85 (1)	C4—C5	1.375 (3)
O3W—H3W2	0.86 (1)	C4—H4	0.9300
N1—C2	1.332 (2)	C5—H5	0.9300
N1—C5	1.345 (3)	C6—C7	1.498 (3)
N2—C4	1.332 (3)	C7—C8	1.421 (2)
N2—C3	1.344 (3)	C9—C10	1.374 (3)
N3—C3	1.339 (3)	C9—H9	0.9300
N3—H31	0.854 (10)	C10—H10	0.9300
N3—H32	0.858 (10)	C11—C12	1.511 (2)
N4—C7	1.335 (2)	C12—C13	1.426 (2)
N4—C10	1.339 (2)	C14—C15	1.377 (3)
N5—C9	1.326 (3)	C14—H14	0.9300
N5—C8	1.347 (3)	C15—H15	0.9300
O1—Ni1—O3	93.69 (6)	H103—N10—H104	111.9 (14)
O1—Ni1—O5	169.25 (5)	O2—C1—O1	125.01 (18)
O3—Ni1—O5	94.48 (6)	O2—C1—C2	118.53 (16)
O1—Ni1—N7	93.74 (6)	O1—C1—C2	116.43 (16)
O3—Ni1—N7	86.83 (5)	N1—C2—C3	120.28 (17)
O5—Ni1—N7	79.76 (5)	N1—C2—C1	115.18 (16)
O1—Ni1—N4	93.66 (6)	C3—C2—C1	124.54 (17)
O3—Ni1—N4	79.14 (5)	N3—C3—N2	118.03 (19)
O5—Ni1—N4	94.73 (6)	N3—C3—C2	121.75 (19)
N7—Ni1—N4	164.51 (6)	N2—C3—C2	120.16 (18)
O1—Ni1—N1	79.56 (6)	N2—C4—C5	123.1 (2)
O3—Ni1—N1	170.67 (6)	N2—C4—H4	118.5
O5—Ni1—N1	93.05 (6)	C5—C4—H4	118.5
N7—Ni1—N1	99.95 (6)	N1—C5—C4	119.79 (19)
N4—Ni1—N1	94.76 (6)	N1—C5—H5	120.1
C1—O1—Ni1	115.52 (12)	C4—C5—H5	120.1
C6—O3—Ni1	116.28 (12)	O4—C6—O3	124.70 (18)
C11—O5—Ni1	115.38 (11)	O4—C6—C7	119.59 (17)
H1W1—O1W—H1W2	111 (2)	O3—C6—C7	115.71 (16)
H2W1—O2W—H2W2	111 (2)	N4—C7—C8	120.49 (17)
H3W1—O3W—H3W2	108 (2)	N4—C7—C6	115.85 (15)
C2—N1—C5	119.07 (16)	C8—C7—C6	123.65 (17)
C2—N1—Ni1	111.95 (12)	N6—C8—N5	117.76 (17)
C5—N1—Ni1	127.91 (13)	N6—C8—C7	122.34 (18)
C4—N2—C3	117.56 (18)	N5—C8—C7	119.90 (17)
C3—N3—H31	117.1 (19)	N5—C9—C10	123.55 (18)
C3—N3—H32	118 (2)	N5—C9—H9	118.2
H31—N3—H32	124 (3)	C10—C9—H9	118.2
C7—N4—C10	119.01 (15)	N4—C10—C9	119.64 (18)
C7—N4—Ni1	112.91 (12)	N4—C10—H10	120.2
C10—N4—Ni1	128.06 (13)	C9—C10—H10	120.2
C9—N5—C8	117.40 (16)	O6—C11—O5	125.35 (17)
C8—N6—H61	114.4 (17)	O6—C11—C12	119.08 (16)

C8—N6—H62	120.9 (18)	O5—C11—C12	115.57 (16)
H61—N6—H62	125 (2)	N7—C12—C13	120.69 (15)
C12—N7—C15	119.61 (15)	N7—C12—C11	115.47 (15)
C12—N7—Ni1	113.37 (11)	C13—C12—C11	123.71 (16)
C15—N7—Ni1	125.84 (12)	N9—C13—N8	117.77 (16)
C14—N8—C13	117.41 (16)	N9—C13—C12	122.88 (16)
C13—N9—H91	116.9 (18)	N8—C13—C12	119.35 (16)
C13—N9—H92	120.5 (17)	N8—C14—C15	123.63 (17)
H91—N9—H92	121 (2)	N8—C14—H14	118.2
H101—N10—H102	107.1 (13)	C15—C14—H14	118.2
H101—N10—H103	108.6 (14)	N7—C15—C14	119.21 (17)
H102—N10—H103	108.8 (13)	N7—C15—H15	120.4
H101—N10—H104	110.0 (13)	C14—C15—H15	120.4
H102—N10—H104	110.3 (14)		
O3—Ni1—O1—C1	170.89 (13)	O1—C1—C2—C3	−167.48 (17)
O5—Ni1—O1—C1	−49.7 (3)	C4—N2—C3—N3	177.4 (2)
N7—Ni1—O1—C1	−102.05 (13)	C4—N2—C3—C2	0.1 (3)
N4—Ni1—O1—C1	91.56 (13)	N1—C2—C3—N3	−175.23 (18)
N1—Ni1—O1—C1	−2.61 (13)	C1—C2—C3—N3	3.6 (3)
O1—Ni1—O3—C6	−96.17 (15)	N1—C2—C3—N2	2.0 (3)
O5—Ni1—O3—C6	90.82 (15)	C1—C2—C3—N2	−179.12 (17)
N7—Ni1—O3—C6	170.28 (15)	C3—N2—C4—C5	−1.0 (3)
N4—Ni1—O3—C6	−3.15 (14)	C2—N1—C5—C4	2.1 (3)
O1—Ni1—O5—C11	−53.7 (3)	Ni1—N1—C5—C4	−164.99 (16)
O3—Ni1—O5—C11	85.68 (13)	N2—C4—C5—N1	0.0 (4)
N7—Ni1—O5—C11	−0.27 (13)	Ni1—O3—C6—O4	−177.33 (18)
N4—Ni1—O5—C11	165.13 (13)	Ni1—O3—C6—C7	3.5 (2)
N1—Ni1—O5—C11	−99.84 (13)	C10—N4—C7—C8	−0.6 (3)
O1—Ni1—N1—C2	8.76 (12)	Ni1—N4—C7—C8	178.20 (13)
O5—Ni1—N1—C2	−179.11 (12)	C10—N4—C7—C6	−179.78 (17)
N7—Ni1—N1—C2	100.76 (12)	Ni1—N4—C7—C6	−1.0 (2)
N4—Ni1—N1—C2	−84.10 (13)	O4—C6—C7—N4	179.16 (19)
O1—Ni1—N1—C5	176.63 (17)	O3—C6—C7—N4	−1.6 (3)
O5—Ni1—N1—C5	−11.24 (17)	O4—C6—C7—C8	0.0 (3)
N7—Ni1—N1—C5	−91.37 (17)	O3—C6—C7—C8	179.19 (17)
N4—Ni1—N1—C5	83.77 (17)	C9—N5—C8—N6	178.60 (19)
O1—Ni1—N4—C7	95.17 (13)	C9—N5—C8—C7	−1.0 (3)
O3—Ni1—N4—C7	2.10 (12)	N4—C7—C8—N6	−178.67 (19)
O5—Ni1—N4—C7	−91.56 (13)	C6—C7—C8—N6	0.5 (3)
N7—Ni1—N4—C7	−23.2 (3)	N4—C7—C8—N5	0.9 (3)
N1—Ni1—N4—C7	174.97 (12)	C6—C7—C8—N5	−179.97 (17)
O1—Ni1—N4—C10	−86.20 (16)	C8—N5—C9—C10	0.8 (3)
O3—Ni1—N4—C10	−179.26 (17)	C7—N4—C10—C9	0.4 (3)
O5—Ni1—N4—C10	87.08 (16)	Ni1—N4—C10—C9	−178.17 (14)
N7—Ni1—N4—C10	155.4 (2)	N5—C9—C10—N4	−0.5 (3)
N1—Ni1—N4—C10	−6.39 (17)	Ni1—O5—C11—O6	177.70 (16)
O1—Ni1—N7—C12	175.68 (12)	Ni1—O5—C11—C12	−3.4 (2)
O3—Ni1—N7—C12	−90.82 (12)	C15—N7—C12—C13	0.5 (2)
O5—Ni1—N7—C12	4.31 (12)	Ni1—N7—C12—C13	168.81 (12)

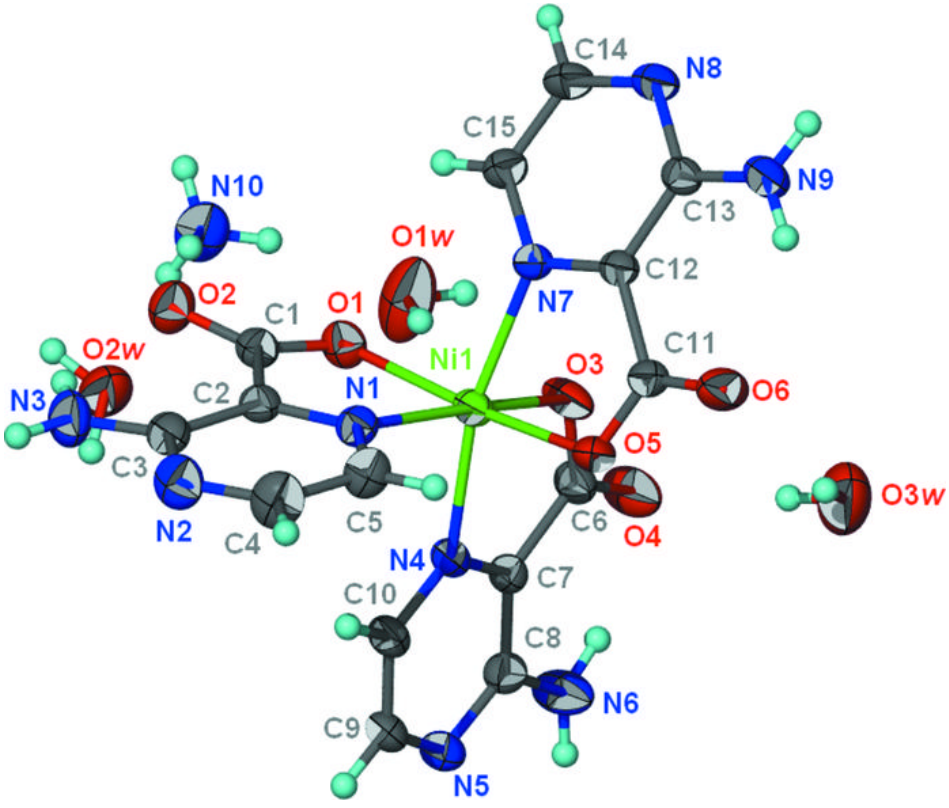
N4—Ni1—N7—C12	−65.9 (3)	C15—N7—C12—C11	−175.57 (16)
N1—Ni1—N7—C12	95.64 (12)	Ni1—N7—C12—C11	−7.23 (18)
O1—Ni1—N7—C15	−16.83 (15)	O6—C11—C12—N7	−173.77 (17)
O3—Ni1—N7—C15	76.67 (15)	O5—C11—C12—N7	7.2 (2)
O5—Ni1—N7—C15	171.80 (16)	O6—C11—C12—C13	10.3 (3)
N4—Ni1—N7—C15	101.6 (2)	O5—C11—C12—C13	−168.66 (16)
N1—Ni1—N7—C15	−96.87 (15)	C14—N8—C13—N9	−177.44 (18)
Ni1—O1—C1—O2	174.82 (15)	C14—N8—C13—C12	2.5 (3)
Ni1—O1—C1—C2	−3.5 (2)	N7—C12—C13—N9	177.08 (17)
C5—N1—C2—C3	−3.1 (3)	C11—C12—C13—N9	−7.2 (3)
Ni1—N1—C2—C3	166.01 (13)	N7—C12—C13—N8	−2.9 (2)
C5—N1—C2—C1	177.96 (16)	C11—C12—C13—N8	172.81 (16)
Ni1—N1—C2—C1	−12.97 (18)	C13—N8—C14—C15	0.1 (3)
O2—C1—C2—N1	−166.99 (16)	C12—N7—C15—C14	2.1 (3)
O1—C1—C2—N1	11.5 (2)	Ni1—N7—C15—C14	−164.65 (14)
O2—C1—C2—C3	14.1 (3)	N8—C14—C15—N7	−2.5 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H31···O2	0.85 (1)	2.08 (2)	2.740 (3)	134 (2)
N6—H61···O4	0.85 (1)	2.01 (2)	2.701 (3)	138 (2)
N6—H62···N5^{i}	0.85 (1)	2.15 (1)	2.992 (2)	175 (2)
N9—H91···O6	0.86 (1)	2.07 (2)	2.733 (2)	134 (2)
N9—H92···O3^{ii}	0.85 (1)	2.10 (1)	2.924 (2)	164 (2)
N10—H101···O2	0.86 (1)	1.91 (1)	2.756 (3)	169 (3)
N10—H102···O1w	0.86 (1)	1.94 (1)	2.779 (3)	164 (3)
N10—H103···O2w	0.85 (1)	2.07 (1)	2.919 (3)	172 (3)
N10—H104···N8^{iii}	0.84 (1)	2.36 (2)	3.018 (3)	135 (2)
O1w—H1w1···O1	0.84 (1)	2.25 (2)	2.964 (3)	143 (4)
O1w—H1w2···N2^{iv}	0.85 (1)	2.00 (1)	2.842 (3)	171 (4)
O2w—H2w1···O5^{v}	0.85 (1)	2.03 (1)	2.869 (2)	168 (3)
O2w—H2w2···O6^{vi}	0.84 (1)	1.96 (1)	2.766 (2)	159 (3)
O3w—H3w1···O4	0.85 (1)	2.39 (5)	2.812 (3)	111 (4)

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

Fig. 1


C14
N8
C15
N10
N9
C13
O1w
C12
O2
C1
O1
N7
C11
O2w
Ni1
O3
N3
C2
N1
O6
O5
C3
O3w
C6
N2
C4
C5
O4
N4
C7
C10
C8
N6
C9
N5