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Tetraaquabis(pyridine-κN)nickel(II) dinitrate

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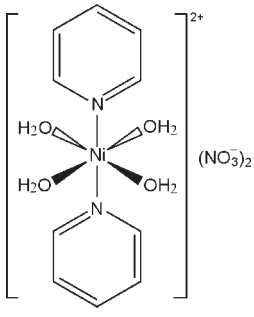
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.049; wR factor = 0.129; data-to-parameter ratio = 21.1.

In the title compound, $[Ni(C_5H_5N)_2(H_2O)_4](NO_3)_2$, the Ni^{II} ion is coordinated by two *N*-bonded pyridine ligands and four water molecules in an octahedral coordination mode. The asymmetric unit consists of one Ni^{II} ion located on an inversion center, as well as one pyridine ligand, one nitrate anion and two water molecules in general positions. In the crystal structure, the discrete complex cations and nitrate anions are connected by O—H···O and C—H···O hydrogen bonds.

Related literature

For general background to thermal decomposition reactions as an alternative tool for the discovery and preparation of new ligand-deficient coordination polymers with defined magnetic properties, see: Wriedt & Näther (2009*a*,*b*); Wriedt *et al.* (2009*a*,*b*). For a related structure, see: Halut-Desportes (1981).



Experimental

Crystal data

$[Ni(C_5H_5N)_2(H_2O)_4](NO_3)_2$
M_r = 412.99
Monoclinic, $P2_1/n$
a = 7.3245 (4) Å
b = 11.3179 (6) Å
c = 10.9347 (5) Å
β = 96.436 (4)°
V = 900.75 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.13 mm^{-1}
T = 293 K
0.28 × 0.16 × 0.07 mm

Data collection

Stoe IPDS-2 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.801, T_{max} = 0.927
12828 measured reflections
2427 independent reflections
2087 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.129
S = 1.15
2427 reflections
115 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O4	2.113 (2)	Ni1—N1	2.140 (2)
Ni1—O5	2.128 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H1*O4*···O2^{i}	0.82	2.39	3.209 (4)	174
O4—H2*O4*···O1^{ii}	0.82	2.26	3.077 (4)	179
O4—H3*O4*···O1	0.82	2.32	3.087 (3)	157
O5—H1*O5*···O3^{iii}	0.82	2.28	3.091 (4)	169
O5—H2*O5*···O1	0.82	2.43	3.191 (4)	155
C2—H2···O1^{iv}	0.93	2.50	3.310 (4)	145
C4—H4···O2^{v}	0.93	2.54	3.461 (4)	170

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $-x+2, -y+1, -z+1$; (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) $-x+\frac{5}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: HY2315).

References

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supporting information

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Tetraaquabis(pyridine-κN)nickel(II) dinitrate

Mario Wriedt, Inke Jess and Christian Näther

S1. Comment

Recently, we have shown that thermal decomposition reactions are an elegant route for the discovery and preparation of new ligand-deficient coordination polymers with defined magnetic properties (Wriedt & Näther, 2009a,b; Wriedt *et al.*, 2009a,b). In our ongoing investigation on the synthesis, structures and properties of such compounds based on paramagnetic transition metal pseudo-halides and N-donor ligands, we have reacted nickel(II) dinitrate hexahydrate, sodium dicyanamide and pyridine. In this reaction single crystals of the title compound were grown.

The title compound (Fig. 1) represents a discrete complex cation, in which the Ni^{II} atom, lying on an inversion center, is coordinated by two pyridine ligands and four water molecules in an octahedral coordination mode. The nitrate anions are not coordinated to the metal atoms (Fig. 2). The NiN_2O_4 octahedron is slightly distorted with Ni—$N_{pyridine}$ distances of 2.140 (2) Å and Ni—O_{water} distances of 2.113 (2) and 2.128 (2) Å (Table 1). The angles arround the metal atoms range between 85.71 (10) to 94.29 (10) and 180°. A similar coordination is found in a related structure (Halut-Desportes, 1981). The opposite pyridyl rings are coplanar due to symmetry. The shortest intermolecular Ni···Ni distance amounts to 7.3245 (4) Å.

S2. Experimental

$Ni(NO_3)_2.6H_2O$ (72.7 mg, 0.25 mmol), sodium dicyanamide (44.5 mg, 0.5 mmol) and pyridine (0.5 ml) obtained from Alfa Aesar were reacted in a closed test-tube at 120°C for 3 d. On cooling light green block-shaped single crystals of the title compound were grown in a mixture with unknown phases.

S3. Refinement

All H atoms were located in a difference Fourier map. H atoms bound to C atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$. The water H atoms were disordered over three positions for each water molecule and were refined as riding, with O—H = 0.82 Å and $U_{iso}(H) = 1.5U_{eq}(O)$, using a split model with SOF = 0.6667 for each H atom.

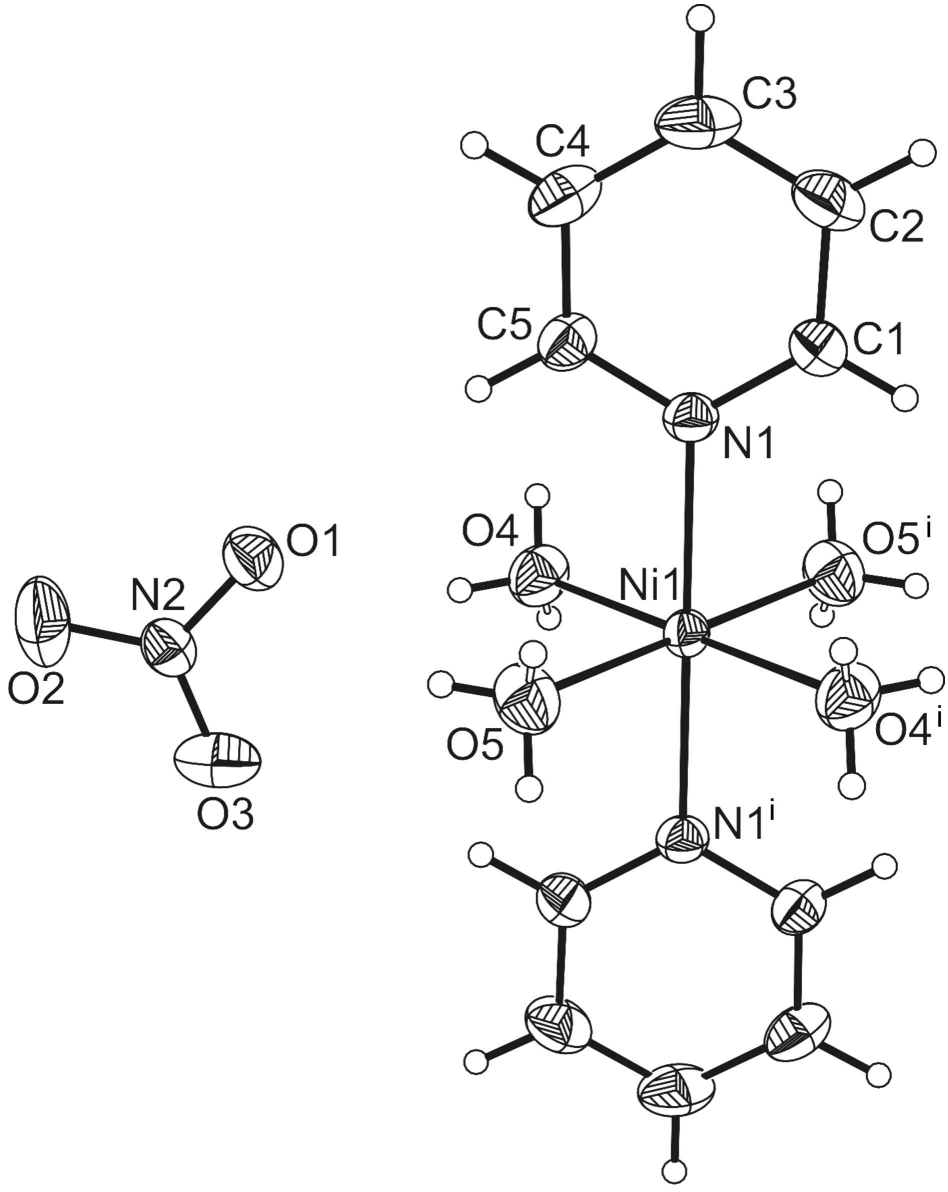

Figure 1

The structure of the title compound with displacement ellipsoids drawn at the 30% probability level. Disordering of the H atoms is shown with full and open bonds. [Symmetry code: (i) -x+1, -y+1, -z+1.]

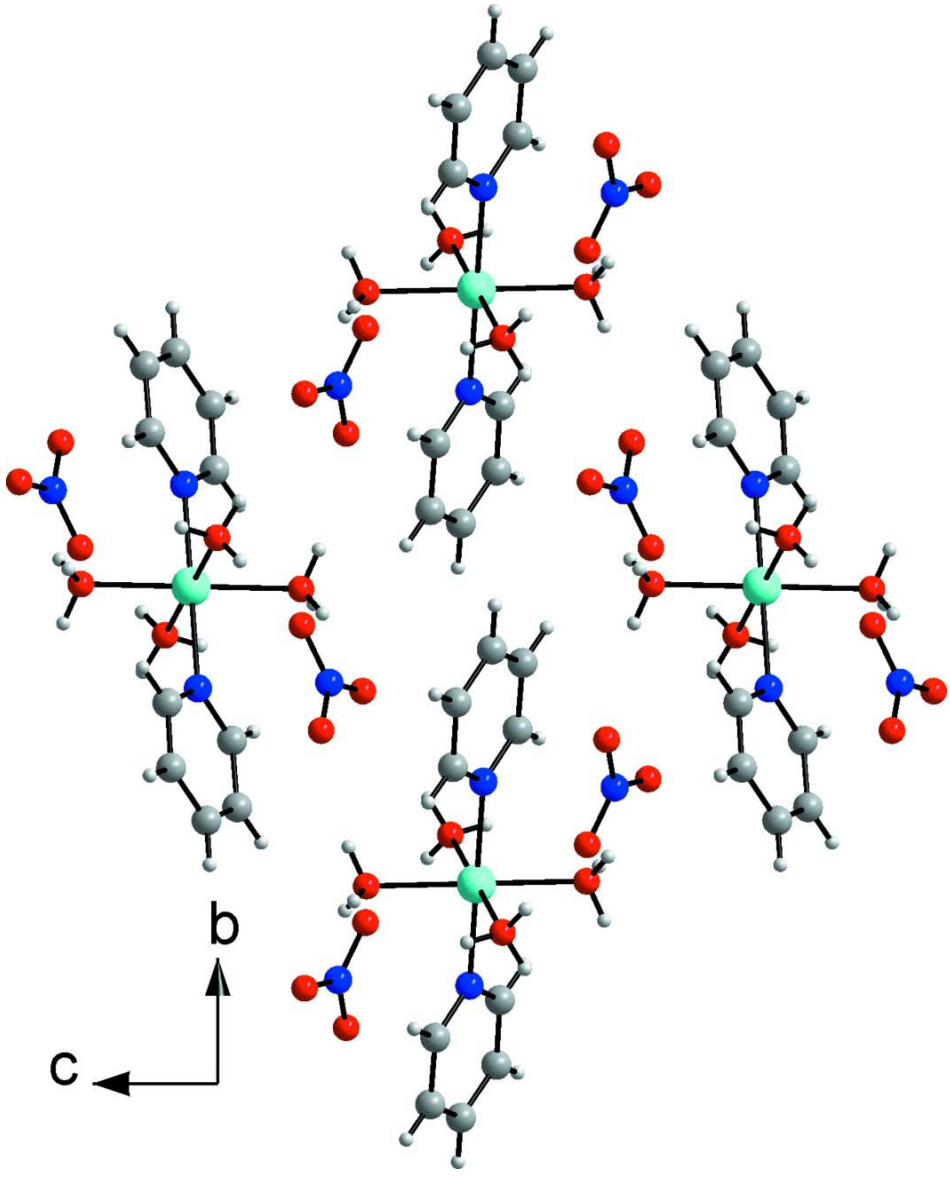

Figure 2

Packing arrangement of the title compound with view along the *a* axis.

Tetraaquabis(pyridine-κ*N*)nickel(II) dinitrate

Crystal data

$[Ni(C_5H_5N)_2(H_2O)_4](NO_3)_2$
$M_r = 412.99$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.3245\ (4)$ Å
$b = 11.3179\ (6)$ Å
$c = 10.9347\ (5)$ Å
$\beta = 96.436\ (4)°$
$V = 900.75\ (8)$ Å^3
$Z = 2$
$F(000) = 428$
$D_x = 1.523$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 12828 reflections
$\theta = 2.6–29.2°$
$\mu = 1.13$ mm^{-1}
$T = 293$ K
Block, light green
0.28 × 0.16 × 0.07 mm

Data collection

Stoe IPDS-2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.801, T_{max} = 0.927
12828 measured reflections
2427 independent reflections
2087 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 29.2°, θ_{min} = 2.6°
h = −10→9
k = −15→15
l = −15→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.129
S = 1.15
2427 reflections
115 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
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.6589P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ni1	0.5000	0.5000	0.5000	0.03618 (15)	
N1	0.6201 (3)	0.32862 (19)	0.4879 (2)	0.0421 (5)	
C1	0.5276 (4)	0.2407 (3)	0.4281 (3)	0.0526 (7)	
H1	0.4095	0.2555	0.3908	0.063*	
C2	0.5994 (6)	0.1285 (3)	0.4191 (4)	0.0678 (9)	
H2	0.5305	0.0693	0.3770	0.081*	
C3	0.7731 (6)	0.1062 (3)	0.4729 (4)	0.0735 (10)	
H3	0.8250	0.0317	0.4676	0.088*	
C4	0.8695 (4)	0.1951 (3)	0.5348 (4)	0.0629 (8)	
H4	0.9881	0.1819	0.5722	0.076*	
C5	0.7896 (4)	0.3040 (3)	0.5412 (3)	0.0486 (6)	
H5	0.8560	0.3636	0.5844	0.058*	
N2	1.0498 (3)	0.6620 (2)	0.7423 (2)	0.0501 (5)	
O1	1.0070 (3)	0.5655 (2)	0.6942 (2)	0.0635 (6)	
O2	1.1995 (4)	0.6760 (3)	0.8009 (3)	0.0956 (10)	
O3	0.9347 (5)	0.7424 (3)	0.7281 (3)	0.0905 (9)	
O4	0.7425 (3)	0.5820 (2)	0.4544 (2)	0.0624 (6)	
H1O4	0.7224	0.6435	0.4154	0.094*	0.667
H2O4	0.8088	0.5431	0.4141	0.094*	0.667
H3O4	0.8104	0.5993	0.5166	0.094*	0.667
O5	0.5815 (4)	0.5041 (2)	0.6930 (2)	0.0670 (6)	
H1O5	0.5830	0.4380	0.7239	0.101*	0.667
H2O5	0.6849	0.5295	0.7147	0.101*	0.667
H3O5	0.5148	0.5457	0.7304	0.101*	0.667

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0330 (2)	0.0335 (2)	0.0415 (2)	−0.00085 (16)	0.00174 (15)	−0.00095 (18)
N1	0.0411 (10)	0.0356 (10)	0.0498 (11)	0.0028 (8)	0.0065 (9)	−0.0007 (9)
C1	0.0526 (15)	0.0430 (14)	0.0613 (17)	−0.0008 (12)	0.0017 (13)	−0.0071 (12)
C2	0.083 (2)	0.0423 (16)	0.079 (2)	−0.0013 (15)	0.0110 (18)	−0.0126 (15)
C3	0.080 (2)	0.0451 (17)	0.099 (3)	0.0188 (16)	0.025 (2)	0.0026 (18)
C4	0.0477 (16)	0.0579 (18)	0.085 (2)	0.0131 (14)	0.0137 (15)	0.0159 (17)
C5	0.0403 (13)	0.0465 (14)	0.0595 (16)	0.0008 (11)	0.0069 (11)	0.0062 (12)
N2	0.0541 (13)	0.0526 (14)	0.0443 (11)	−0.0080 (11)	0.0088 (10)	−0.0055 (10)
O1	0.0690 (14)	0.0510 (13)	0.0687 (14)	−0.0078 (10)	−0.0002 (11)	−0.0095 (11)
O2	0.0725 (18)	0.123 (3)	0.0858 (19)	−0.0316 (17)	−0.0137 (15)	−0.0169 (18)
O3	0.104 (2)	0.0689 (17)	0.102 (2)	0.0254 (16)	0.0278 (18)	−0.0123 (15)
O4	0.0518 (12)	0.0568 (13)	0.0788 (15)	−0.0035 (10)	0.0088 (10)	0.0059 (11)
O5	0.0763 (16)	0.0655 (15)	0.0575 (13)	−0.0031 (11)	0.0000 (11)	−0.0016 (11)

Geometric parameters (Å, º)

Ni1—O4	2.113 (2)	C4—H4	0.9300
Ni1—O5	2.128 (2)	C5—H5	0.9300
Ni1—N1	2.140 (2)	N2—O2	1.216 (4)
N1—C1	1.333 (4)	N2—O1	1.238 (3)
N1—C5	1.340 (3)	N2—O3	1.238 (4)
C1—C2	1.381 (4)	O4—H1O4	0.8200
C1—H1	0.9300	O4—H2O4	0.8200
C2—C3	1.365 (5)	O4—H3O4	0.8200
C2—H2	0.9300	O5—H1O5	0.8200
C3—C4	1.364 (5)	O5—H2O5	0.8200
C3—H3	0.9300	O5—H3O5	0.8200
C4—C5	1.369 (4)		
O4—Ni1—O4^{i}	180.00 (11)	C4—C3—C2	118.9 (3)
O4—Ni1—O5^{i}	85.71 (10)	C4—C3—H3	120.6
O4^{i}—Ni1—O5^{i}	94.29 (10)	C2—C3—H3	120.6
O4—Ni1—O5	94.29 (10)	C3—C4—C5	119.3 (3)
O4^{i}—Ni1—O5	85.71 (10)	C3—C4—H4	120.4
O5^{i}—Ni1—O5	180.000 (1)	C5—C4—H4	120.4
O4—Ni1—N1	91.23 (9)	N1—C5—C4	123.1 (3)
O4^{i}—Ni1—N1	88.77 (9)	N1—C5—H5	118.5
O5^{i}—Ni1—N1	89.46 (9)	C4—C5—H5	118.5
O5—Ni1—N1	90.54 (9)	O2—N2—O1	120.5 (3)
O4—Ni1—N1^{i}	88.77 (9)	O2—N2—O3	122.1 (3)
O4^{i}—Ni1—N1^{i}	91.23 (9)	O1—N2—O3	117.3 (3)
O5^{i}—Ni1—N1^{i}	90.54 (9)	Ni1—O4—H1O4	112.9
O5—Ni1—N1^{i}	89.46 (9)	Ni1—O4—H2O4	117.0
N1—Ni1—N1^{i}	180.000 (1)	H1O4—O4—H2O4	105.0
C1—N1—C5	116.9 (2)	Ni1—O4—H3O4	110.9

C1—N1—Ni1	121.15 (19)	H1O4—O4—H3O4	106.6
C5—N1—Ni1	121.94 (19)	H2O4—O4—H3O4	103.5
N1—C1—C2	123.0 (3)	Ni1—O5—H1O5	112.2
N1—C1—H1	118.5	Ni1—O5—H2O5	116.2
C2—C1—H1	118.5	H1O5—O5—H2O5	103.3
C3—C2—C1	118.9 (3)	Ni1—O5—H3O5	113.0
C3—C2—H2	120.6	H1O5—O5—H3O5	107.4
C1—C2—H2	120.6	H2O5—O5—H3O5	103.7

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H1*O*4···O2^{ii}	0.82	2.39	3.209 (4)	174
O4—H2*O*4···O1^{iii}	0.82	2.26	3.077 (4)	179
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O5—H1*O*5···O3^{iv}	0.82	2.28	3.091 (4)	169
O5—H2*O*5···O1	0.82	2.43	3.191 (4)	155
C2—H2···O1^{v}	0.93	2.50	3.310 (4)	145
C4—H4···O2^{vi}	0.93	2.54	3.461 (4)	170

Symmetry codes: (ii) *x*−1/2, −*y*+3/2, *z*−1/2; (iii) −*x*+2, −*y*+1, −*z*+1; (iv) −*x*+3/2, *y*−1/2, −*z*+3/2; (v) *x*−1/2, −*y*+1/2, *z*−1/2; (vi) −*x*+5/2, *y*−1/2, −*z*+3/2.