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Bis(dicyanamido-κN^1)bis[2-(2-hydroxyethyl)pyridine-$\kappa^2 N,O$]nickel(II)

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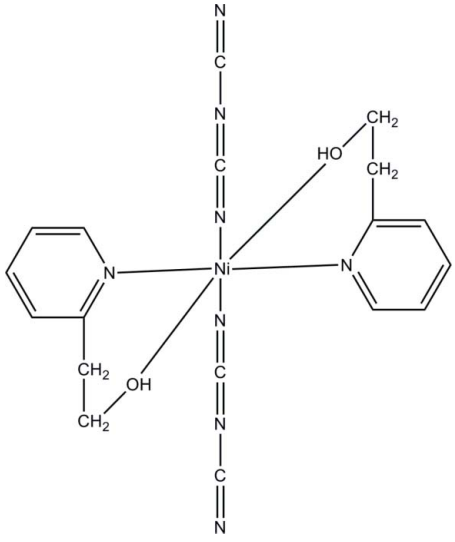
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.034; wR factor = 0.095; data-to-parameter ratio = 12.9.

In the title complex, $[Ni\{N(CN)_2\}_2(C_7H_9NO)_2]$, the Ni^{II} ion (site symmetry $\overline{1}$) adopts a distorted *trans*-NiO_2N_4 octahedral geometry. In the crystal, intermolecular O—H···N hydrogen bonds link the molecules, forming a chain along the c axis.

Related literature

For related structures, see: Boskovic *et al.* (2002); Sanudo *et al.* (2003).



Experimental

Crystal data

$[Ni(C_2N_3)_2(C_7H_9NO)_2]$ M_r = 437.11
Triclinic, $P\overline{1}$
a = 8.1498 (1) Å
b = 8.76020 (11) Å
c = 8.9201 (12) Å
α = 100.841 (1)°
β = 110.588 (2)°
γ = 115.359 (2)°
V = 493.66 (7) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.02 mm^{-1}
T = 298 K
0.28 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.764, T_{max} = 0.863
2566 measured reflections
1718 independent reflections
1579 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.095
S = 1.00
1718 reflections
133 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—N2	2.065 (2)	Ni1—N1	2.095 (2)
Ni1—O1	2.0748 (16)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N4^{i}	0.82	1.89	2.711 (3)	175

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

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5097).

References

Boskovic, C., Brechin, E. K. & Christou, G. (2002). *J. Am. Chem. Soc.* **124**, 3725–3736.
Bruker (2003). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sanudo, E. C., Wernsdorfer, W. & Christou, G. (2003). *Polyhedron*, **22**, 2267–2271.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, m1518 [doi:10.1107/S1600536809043359]

Bis(dicyanamido-κN^1)bis[2-(2-hydroxyethyl)pyridine-$\kappa^2 N,O$]nickel(II)

Ling-Qian Kong, Xiu-Ping Ju and Da-Cheng Li

S1. Comment

In recent years there has been considerable interest in metal complexes supported by hydroxyethyl-pyridine,the ligand due to its versatile coordination activities and bridging function. (Sanudo *et al.*, 2003; Boskovic *et al.*, 2002). As an extension of this work, we have synthesized the title compound, (I), and report herein its crystal structure.

The complex (Fig. 1) consists of two L2-(*L* = (hydroxyethyl)(pyridine)) ligands, one Ni^{II} ion and two dicyanmiden ligands. The coordination geometry around the Ni center is octahedral with a NiN_4O_2 ligand set (Table 1). Two atoms N1 of hydroxyethylpyridine ligand occpy the axial sites. In the crystal structure, intermolecular O—H···N hydrogen bonds link molecules to form a one-dimensional chain along to the *c* axis (Table 2).

S2. Experimental

2-Hydroxyethylpyridine (0.123 g, 1 mmol) was deprotonated by Et_4NOH (25%) in the prensence of nickel nitrate hexahydrate (0.5 mmol, 0.127 g) in a mixture of methanol and acetonitrile (V/V = 1:1) after the solution was stirred at room temperature for 0.5 h. Sodium dicyanmiden (5 mmol 0.486 g) was added to the above solution and then further stirred for 1 h. The resulting clear solution was filtered and left to stand at room temperature. Green blocks of (I) were obtained by slow evaporation of the solvents within 2 weeks. MP = 518-520 K (decomp).

S3. Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms with C—H = 0.93–0.97Å [U_{iso}(H) = 1.2U_{eq}(C)] and O—H = 0.82 Å [U_{iso}(H) = 1.5U_{eq}(O)].

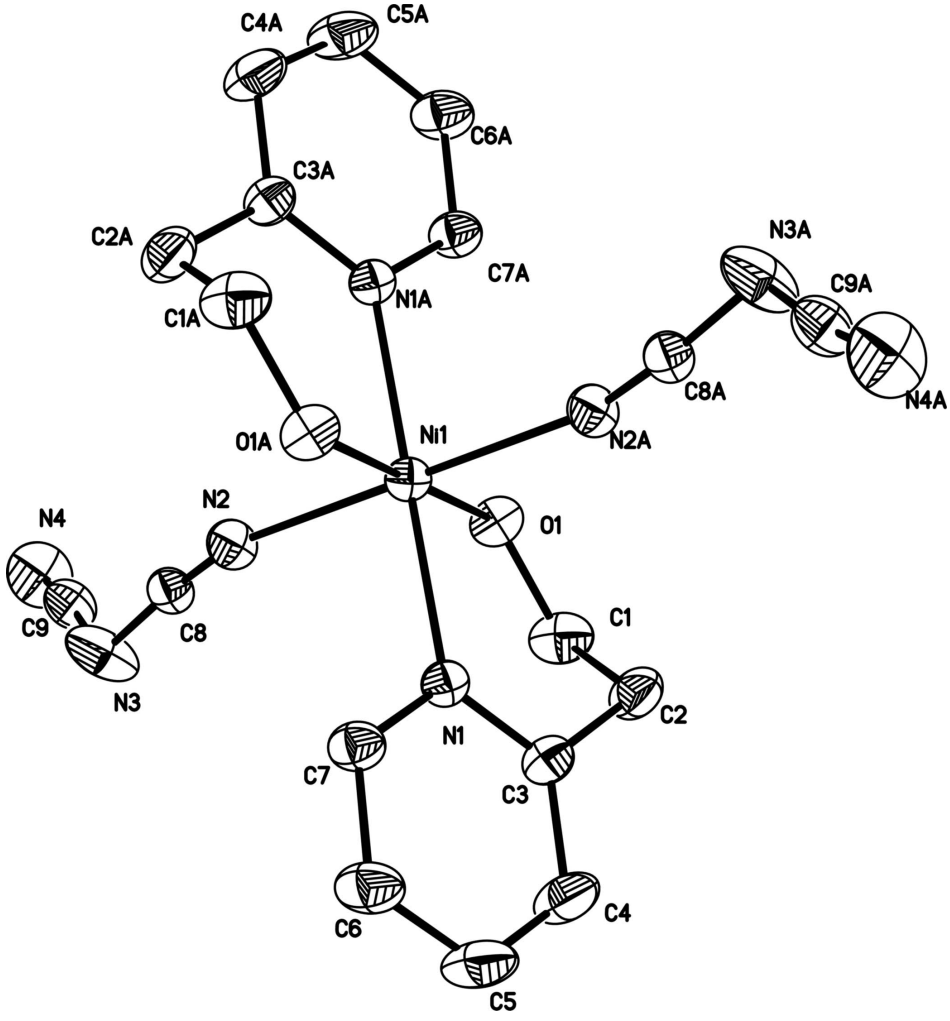


Figure 1

The structure of the title complex, showing 30% probability displacement ellipsoids. Atoms labelled with the suffix A are generated by the symmetry operation (-*x* + 1,-*y*,-*z* + 1). H atoms have been omitted for clarity.

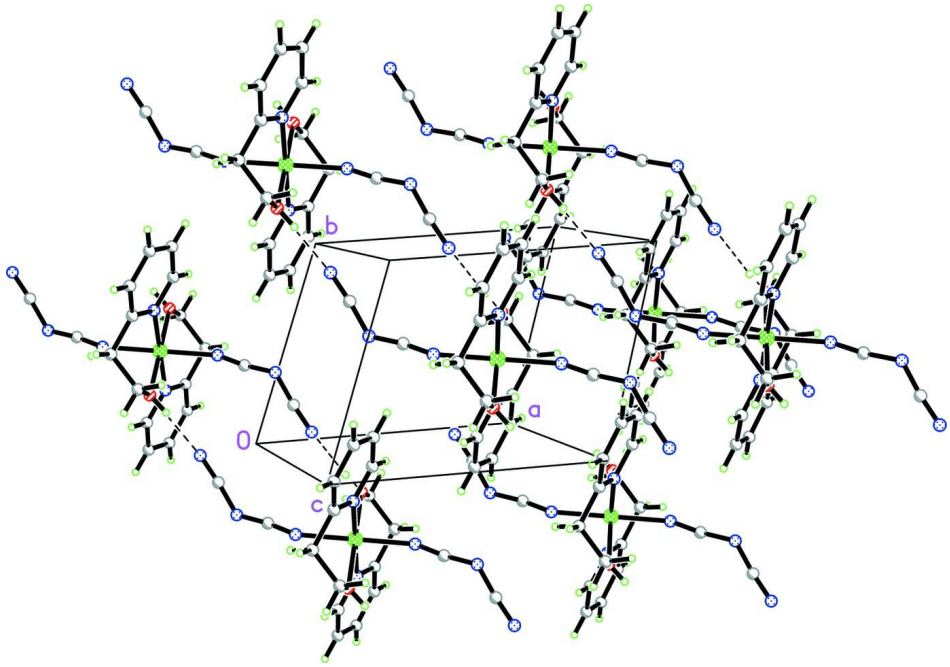


Figure 2

The crystal packing of (I), viewed approximately along the *c* axis.

Bis(dicyanamido-κN^1)bis[2-(2-hydroxyethyl)pyridine- $\kappa^2 N,O$]nickel(II)

Crystal data

$[Ni(C_2N_3)_2(C_7H_9NO)_2]$
$M_r = 437.11$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.1498$ (1) Å
$b = 8.76020$ (11) Å
$c = 8.9201$ (12) Å
$\alpha = 100.841$ (1)°
$\beta = 110.588$ (2)°
$\gamma = 115.359$ (2)°
$V = 493.66$ (7) Å^3
$Z = 1$
$F(000) = 226$
$D_x = 1.47$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1464 reflections
$\theta = 2.7$–26.3°
$\mu = 1.02$ mm^{-1}
$T = 298$ K
Block, green
0.28 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
$T_{min} = 0.764$, $T_{max} = 0.863$
2566 measured reflections
1718 independent reflections
1579 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.7°$
$h = -9 \rightarrow 9$
$k = -7 \rightarrow 10$
$l = -10 \rightarrow 8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.095$
$S = 1.00$
1718 reflections
133 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
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.0746P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.5000	0.0000	0.5000	0.03081 (18)
N1	0.7472 (3)	0.2759 (3)	0.6426 (3)	0.0341 (5)
N2	0.4639 (3)	0.0453 (3)	0.2748 (3)	0.0415 (5)
N3	0.3758 (4)	0.1688 (4)	0.0579 (4)	0.0700 (9)
N4	0.0438 (5)	0.0438 (4)	−0.1949 (4)	0.0731 (9)
O1	0.2970 (3)	0.0824 (2)	0.5031 (2)	0.0411 (4)
H1	0.1971	0.0414	0.4067	0.062*
C1	0.3611 (5)	0.2644 (4)	0.6012 (4)	0.0548 (8)
H1A	0.2523	0.2612	0.6245	0.066*
H1B	0.3849	0.3390	0.5343	0.066*
C2	0.5577 (5)	0.3491 (4)	0.7713 (4)	0.0537 (8)
H2A	0.5771	0.4562	0.8495	0.064*
H2B	0.5422	0.2608	0.8249	0.064*
C3	0.7468 (4)	0.4061 (4)	0.7512 (3)	0.0418 (6)
C4	0.9168 (5)	0.5852 (4)	0.8400 (4)	0.0614 (9)
H4	0.9143	0.6738	0.9136	0.074*
C5	1.0883 (5)	0.6328 (4)	0.8202 (5)	0.0653 (9)
H5	1.2039	0.7528	0.8817	0.078*
C6	1.0887 (5)	0.5030 (4)	0.7096 (4)	0.0559 (8)
H6	1.2034	0.5326	0.6932	0.067*
C7	0.9155 (4)	0.3274 (4)	0.6227 (4)	0.0428 (6)
H7	0.9151	0.2392	0.5456	0.051*
C8	0.4109 (4)	0.0950 (4)	0.1684 (3)	0.0375 (6)
C9	0.1941 (5)	0.0937 (4)	−0.0759 (4)	0.0489 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0250 (3)	0.0319 (3)	0.0300 (3)	0.0149 (2)	0.00897 (19)	0.01179 (19)
N1	0.0305 (11)	0.0347 (11)	0.0322 (10)	0.0171 (9)	0.0116 (9)	0.0121 (9)
N2	0.0354 (12)	0.0455 (13)	0.0324 (11)	0.0182 (11)	0.0097 (10)	0.0168 (10)
N3	0.0383 (14)	0.0750 (19)	0.0627 (17)	0.0120 (13)	0.0049 (13)	0.0479 (16)
N4	0.0540 (17)	0.096 (2)	0.0586 (17)	0.0432 (17)	0.0088 (15)	0.0389 (17)
O1	0.0317 (9)	0.0441 (10)	0.0426 (10)	0.0243 (8)	0.0104 (8)	0.0135 (8)
C1	0.0432 (16)	0.0479 (17)	0.079 (2)	0.0307 (14)	0.0288 (16)	0.0222 (16)
C2	0.0537 (18)	0.0443 (16)	0.0553 (18)	0.0233 (15)	0.0298 (15)	0.0066 (14)
C3	0.0383 (14)	0.0379 (14)	0.0383 (14)	0.0186 (12)	0.0138 (12)	0.0090 (12)
C4	0.055 (2)	0.0382 (16)	0.065 (2)	0.0180 (15)	0.0240 (17)	0.0000 (15)
C5	0.0440 (18)	0.0349 (16)	0.084 (2)	0.0086 (14)	0.0234 (17)	0.0074 (16)
C6	0.0350 (15)	0.0443 (16)	0.072 (2)	0.0136 (13)	0.0231 (15)	0.0168 (15)
C7	0.0343 (14)	0.0380 (14)	0.0502 (16)	0.0177 (12)	0.0188 (13)	0.0140 (13)
C8	0.0251 (12)	0.0406 (14)	0.0343 (13)	0.0117 (11)	0.0108 (11)	0.0133 (12)
C9	0.0486 (17)	0.0551 (17)	0.0520 (17)	0.0306 (15)	0.0239 (15)	0.0326 (15)

Geometric parameters (Å, º)

Ni1—N2^{i}	2.065 (2)	C1—C2	1.506 (4)
Ni1—N2	2.065 (2)	C1—H1A	0.9700
Ni1—O1	2.0748 (16)	C1—H1B	0.9700
Ni1—O1^{i}	2.0748 (16)	C2—C3	1.491 (4)
Ni1—N1	2.095 (2)	C2—H2A	0.9700
Ni1—N1^{i}	2.095 (2)	C2—H2B	0.9700
N1—C7	1.337 (3)	C3—C4	1.381 (4)
N1—C3	1.353 (3)	C4—C5	1.365 (5)
N2—C8	1.142 (3)	C4—H4	0.9300
N3—C8	1.293 (3)	C5—C6	1.360 (5)
N3—C9	1.296 (4)	C5—H5	0.9300
N4—C9	1.127 (4)	C6—C7	1.372 (4)
O1—C1	1.422 (3)	C6—H6	0.9300
O1—H1	0.8200	C7—H7	0.9300
N2^{i}—Ni1—N2	180.0	O1—C1—H1B	109.6
N2^{i}—Ni1—O1	92.61 (8)	C2—C1—H1B	109.6
N2—Ni1—O1	87.39 (8)	H1A—C1—H1B	108.1
N2^{i}—Ni1—O1^{i}	87.39 (8)	C3—C2—C1	113.5 (3)
N2—Ni1—O1^{i}	92.61 (8)	C3—C2—H2A	108.9
O1—Ni1—O1^{i}	180.0	C1—C2—H2A	108.9
N2^{i}—Ni1—N1	91.92 (8)	C3—C2—H2B	108.9
N2—Ni1—N1	88.08 (8)	C1—C2—H2B	108.9
O1—Ni1—N1	89.07 (7)	H2A—C2—H2B	107.7
O1^{i}—Ni1—N1	90.93 (7)	N1—C3—C4	120.6 (3)
N2^{i}—Ni1—N1^{i}	88.08 (8)	N1—C3—C2	117.7 (2)
N2—Ni1—N1^{i}	91.92 (8)	C4—C3—C2	121.7 (3)

O1—Ni1—N1^{i}	90.93 (7)	C5—C4—C3	120.3 (3)
O1^{i}—Ni1—N1^{i}	89.07 (7)	C5—C4—H4	119.8
N1—Ni1—N1^{i}	180.0	C3—C4—H4	119.8
C7—N1—C3	117.8 (2)	C6—C5—C4	119.4 (3)
C7—N1—Ni1	117.74 (17)	C6—C5—H5	120.3
C3—N1—Ni1	124.45 (18)	C4—C5—H5	120.3
C8—N2—Ni1	156.7 (2)	C5—C6—C7	118.3 (3)
C8—N3—C9	122.3 (3)	C5—C6—H6	120.8
C1—O1—Ni1	124.15 (16)	C7—C6—H6	120.8
C1—O1—H1	109.5	N1—C7—C6	123.6 (3)
Ni1—O1—H1	113.9	N1—C7—H7	118.2
O1—C1—C2	110.2 (2)	C6—C7—H7	118.2
O1—C1—H1A	109.6	N2—C8—N3	172.5 (3)
C2—C1—H1A	109.6	N4—C9—N3	173.2 (3)
N2^{i}—Ni1—N1—C7	115.4 (2)	O1—C1—C2—C3	74.7 (3)
N2—Ni1—N1—C7	−64.6 (2)	C7—N1—C3—C4	−1.0 (4)
O1—Ni1—N1—C7	−152.00 (19)	Ni1—N1—C3—C4	−179.8 (2)
O1^{i}—Ni1—N1—C7	28.00 (19)	C7—N1—C3—C2	179.1 (3)
N2^{i}—Ni1—N1—C3	−65.8 (2)	Ni1—N1—C3—C2	0.3 (3)
N2—Ni1—N1—C3	114.2 (2)	C1—C2—C3—N1	−56.9 (4)
O1—Ni1—N1—C3	26.8 (2)	C1—C2—C3—C4	123.2 (3)
O1^{i}—Ni1—N1—C3	−153.2 (2)	N1—C3—C4—C5	−0.5 (5)
O1—Ni1—N2—C8	9.2 (5)	C2—C3—C4—C5	179.4 (3)
O1^{i}—Ni1—N2—C8	−170.8 (5)	C3—C4—C5—C6	1.3 (6)
N1—Ni1—N2—C8	−80.0 (5)	C4—C5—C6—C7	−0.6 (5)
N1^{i}—Ni1—N2—C8	100.0 (5)	C3—N1—C7—C6	1.8 (4)
N2^{i}—Ni1—O1—C1	84.2 (2)	Ni1—N1—C7—C6	−179.3 (2)
N2—Ni1—O1—C1	−95.8 (2)	C5—C6—C7—N1	−1.0 (5)
N1—Ni1—O1—C1	−7.7 (2)	Ni1—N2—C8—N3	104 (2)
N1^{i}—Ni1—O1—C1	172.3 (2)	C9—N3—C8—N2	177 (2)
Ni1—O1—C1—C2	−34.9 (3)	C8—N3—C9—N4	172 (3)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N4^{ii}	0.82	1.89	2.711 (3)	175

Symmetry code: (ii) −*x*, −*y*, −*z*.