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Bis(acetato-κ^2O,O')bis(2-aminopyridine-κN)nickel(II)

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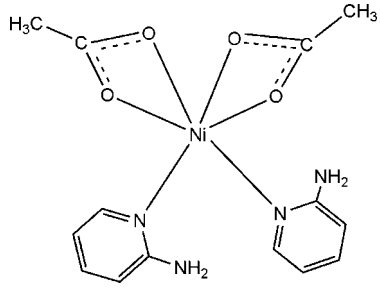
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.031; wR factor = 0.093; data-to-parameter ratio = 14.2.

The title complex, $[Ni(C_2H_3O_2)_2(C_5H_6N_2)_2]$, has a distorted octahedral geometry around the Ni atom. Intermolecular and intramolecular N—H···O hydrogen bonds exist in the crystal structure.

Related literature

For general background, see: Roman *et al.* (1995).



Experimental

Crystal data

$[Ni(C_2H_3O_2)_2(C_5H_6N_2)_2]$
M_r = 365.01
Orthorhombic, *Pbca*
a = 14.281 (4) Å
b = 14.989 (5) Å
c = 15.241 (5) Å
V = 3262.5 (18) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.21 mm^{-1}
T = 298 (2) K
0.48 × 0.36 × 0.30 mm

Data collection

Siemens SMART CCD area–detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.593, T_{max} = 0.712
15953 measured reflections
2875 independent reflections
2173 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.093
S = 0.95
2875 reflections
202 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O2^{i}	0.86	2.13	2.988 (3)	179
N2—H2A···O1	0.86	2.20	2.967 (3)	149
N4—H4A···O4	0.86	2.03	2.848 (4)	158

Symmetry code: (i) $x+\frac{1}{2}, y, -z+\frac{1}{2}$.

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: RK2064).

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Bis(acetato-κ^2O,O')bis(2-aminopyridine-κN)nickel(II)

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S1. Comment

A lot of work has been devoted to the study of mixed–ligand complexes because of their key role in biological progress and their properties in areas such as analytical chemistry, catalysis and magnetochemistry (Roman *et al.*, 1995). In this paper, we report the synthesis and crystal structure of the new title nickel complex.

The molecular structure of the title complex, (I), is shown on Fig.1. The Ni atom is in a distorted octahedral geometry [Ni—N = 2.069 (2)–2.079 (2) Å, Ni—O = 2.092 (2)–2.164 (2) Å], coordinated by two N atoms from two 2–amino-pyridine ligands and four O atoms from two acetate groups. The intermolecular and intramolecular N—H···O hydrogen bonds are exist in the crystal structure.

S2. Experimental

Nickel acetate tetrahydrate (2 mmol, 497.7 mg) in absolute ethanol (20 ml) was added dropwise to a absolute ethanol solution (20 ml) of 2–aminopyridine (4 mmol, 376.5 mg). The mixture was heated under reflux with stirring for 4 h. The solution was kept at room temperature for 20 days, after which large green block–shaped crystals of the title complex suitable for *X*–ray diffraction analysis were obtained.

S3. Refinement

All H–atoms were positioned geometrically and refined using a riding model, with C—H (methyl) 0.96 Å, C—H (aromatic) 0.93 Å, *N*—*H*(amino) 0.86 Å, with U_{iso}(H) = 1.2U_{eq}(C, N) and U_{iso}(H) = 1.5U_{eq}(C) for CH_3.

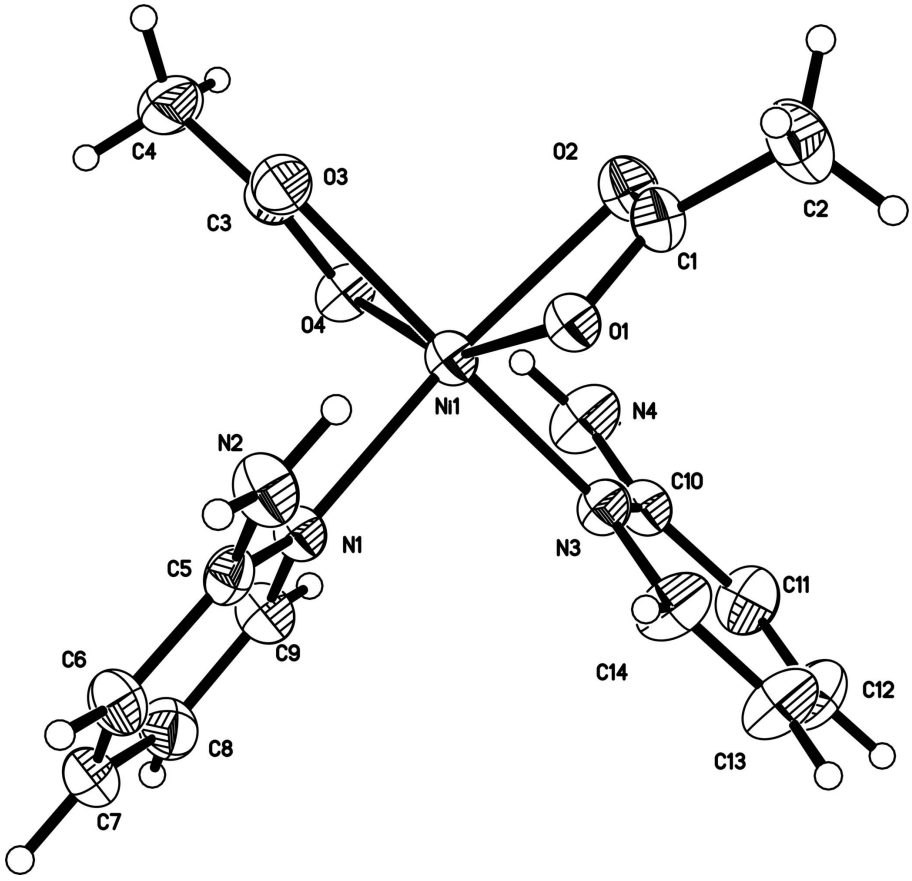

Figure 1

The molecular structure of the title complex with the atom numbering scheme. Displacement ellipsoids are shown at 30% probability level. H atoms are presented as a spheres of arbitrary radius.

Bis(acetato-κ^2O,O')bis(2-aminopyridine-κN)nickel(II)

Crystal data

$[Ni(C_2H_3O_2)_2(C_5H_6N_2)_2]$
$M_r = 365.01$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 14.281\ (4)$ Å
$b = 14.989\ (5)$ Å
$c = 15.241\ (5)$ Å
$V = 3262.5\ (18)$ Å^3
$Z = 8$
$F(000) = 1520$
$D_x = 1.486$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4718 reflections
$\theta = 2.4–27.7°$
$\mu = 1.21$ mm^{-1}
$T = 298$ K
Block, green
0.48 × 0.36 × 0.30 mm

Data collection

Siemens SMART CCD area–detector diffractometer
Radiation source: fine–focus sealed tube
Graphite monochromator
φ– and ω–scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.593$, $T_{max} = 0.712$
15953 measured reflections
2875 independent reflections
2173 reflections with $I > 2\sigma(I)$

R_{int} = 0.039
θ_{max} = 25.0°, θ_{min} = 2.4°
h = −16→16
k = −17→17
l = −14→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.093
S = 0.95
2875 reflections
202 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 4.6901P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 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	0.65501 (2)	0.18431 (2)	0.30692 (2)	0.03492 (14)
N1	0.74523 (16)	0.17626 (15)	0.41287 (15)	0.0353 (6)
N2	0.88178 (18)	0.17679 (18)	0.33118 (18)	0.0475 (7)
H2A	0.8485	0.1789	0.2842	0.057*
H2B	0.9419	0.1759	0.3277	0.057*
N3	0.58868 (17)	0.06682 (16)	0.34427 (17)	0.0405 (6)
N4	0.44625 (19)	0.1315 (2)	0.3738 (2)	0.0654 (9)
H4A	0.4703	0.1828	0.3626	0.079*
H4B	0.3884	0.1274	0.3889	0.079*
O1	0.73266 (14)	0.12566 (15)	0.20530 (14)	0.0469 (5)
O2	0.59071 (15)	0.17241 (14)	0.17892 (14)	0.0470 (5)
O3	0.68659 (16)	0.32137 (14)	0.28166 (15)	0.0484 (6)
O4	0.56826 (15)	0.28132 (14)	0.36282 (15)	0.0472 (5)
C1	0.6643 (2)	0.1341 (2)	0.1533 (2)	0.0470 (5)
C2	0.6716 (3)	0.0967 (3)	0.0625 (3)	0.0723 (12)
H2C	0.6547	0.0348	0.0632	0.108*
H2D	0.7348	0.1029	0.0418	0.108*
H2E	0.6301	0.1286	0.0241	0.108*
C3	0.6159 (2)	0.3419 (2)	0.3261 (2)	0.0445 (8)
C4	0.5880 (3)	0.4383 (2)	0.3375 (3)	0.0667 (11)
H4C	0.6084	0.4591	0.3939	0.100*
H4D	0.5212	0.4435	0.3334	0.100*

H4E	0.6167	0.4736	0.2924	0.100*
C5	0.83976 (19)	0.17483 (18)	0.4102 (2)	0.0364 (7)
C6	0.8921 (2)	0.1720 (2)	0.4878 (2)	0.0500 (8)
H6	0.9572	0.1724	0.4854	0.060*
C7	0.8480 (3)	0.1687 (2)	0.5667 (2)	0.0541 (9)
H7	0.8826	0.1663	0.6184	0.065*
C8	0.7511 (3)	0.1690 (2)	0.5695 (2)	0.0526 (9)
H8	0.7193	0.1665	0.6227	0.063*
C9	0.7040 (2)	0.1731 (2)	0.4926 (2)	0.0458 (8)
H9	0.6389	0.1738	0.4947	0.055*
C10	0.4988 (2)	0.0580 (2)	0.3679 (2)	0.0416 (7)
C11	0.4604 (3)	−0.0261 (2)	0.3863 (3)	0.0609 (10)
H11	0.3981	−0.0312	0.4033	0.073*
C12	0.5143 (3)	−0.0994 (3)	0.3793 (3)	0.0698 (11)
H12	0.4891	−0.1553	0.3910	0.084*
C13	0.6068 (3)	−0.0916 (3)	0.3546 (3)	0.0719 (12)
H13	0.6451	−0.1415	0.3497	0.086*
C14	0.6401 (2)	−0.0087 (2)	0.3377 (3)	0.0608 (10)
H14	0.7023	−0.0034	0.3206	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0245 (2)	0.0418 (2)	0.0384 (2)	−0.00038 (16)	0.00112 (16)	−0.00062 (17)
N1	0.0281 (12)	0.0422 (14)	0.0355 (13)	−0.0029 (10)	0.0029 (10)	−0.0001 (11)
N2	0.0247 (13)	0.0668 (19)	0.0508 (17)	−0.0029 (12)	0.0040 (12)	−0.0038 (14)
N3	0.0295 (13)	0.0412 (14)	0.0508 (15)	−0.0025 (11)	0.0000 (12)	0.0003 (12)
N4	0.0373 (16)	0.0581 (18)	0.101 (3)	0.0006 (14)	0.0157 (17)	0.0063 (18)
O1	0.0317 (11)	0.0616 (14)	0.0475 (13)	0.0035 (10)	−0.0040 (10)	−0.0073 (11)
O2	0.0348 (10)	0.0558 (11)	0.0503 (11)	0.0000 (8)	−0.0056 (8)	−0.0050 (9)
O3	0.0461 (13)	0.0532 (13)	0.0457 (13)	−0.0089 (11)	0.0034 (11)	0.0038 (10)
O4	0.0402 (12)	0.0437 (12)	0.0577 (14)	0.0018 (10)	0.0092 (11)	0.0022 (11)
C1	0.0348 (10)	0.0558 (11)	0.0503 (11)	0.0000 (8)	−0.0056 (8)	−0.0050 (9)
C2	0.078 (3)	0.086 (3)	0.053 (2)	0.013 (2)	−0.012 (2)	−0.023 (2)
C3	0.0465 (19)	0.0452 (18)	0.0419 (18)	0.0012 (15)	−0.0081 (16)	0.0012 (15)
C4	0.091 (3)	0.0416 (19)	0.067 (2)	0.008 (2)	−0.005 (2)	0.0021 (18)
C5	0.0308 (15)	0.0315 (15)	0.0468 (18)	−0.0041 (12)	0.0014 (14)	−0.0025 (13)
C6	0.0346 (18)	0.056 (2)	0.059 (2)	−0.0048 (15)	−0.0110 (16)	−0.0002 (17)
C7	0.059 (2)	0.056 (2)	0.047 (2)	−0.0094 (17)	−0.0172 (18)	0.0030 (16)
C8	0.061 (2)	0.059 (2)	0.0378 (18)	−0.0087 (17)	0.0031 (17)	0.0000 (16)
C9	0.0367 (17)	0.059 (2)	0.0412 (18)	−0.0040 (15)	0.0053 (15)	−0.0003 (15)
C10	0.0330 (16)	0.0505 (18)	0.0414 (17)	−0.0034 (14)	0.0036 (14)	−0.0015 (15)
C11	0.045 (2)	0.063 (2)	0.075 (3)	−0.0148 (18)	0.0148 (19)	0.003 (2)
C12	0.076 (3)	0.048 (2)	0.085 (3)	−0.014 (2)	0.014 (2)	0.005 (2)
C13	0.071 (3)	0.044 (2)	0.101 (3)	0.0033 (19)	0.013 (2)	0.008 (2)
C14	0.043 (2)	0.048 (2)	0.092 (3)	0.0030 (16)	0.0131 (19)	0.011 (2)

Geometric parameters (Å, º)

Ni1—N1	2.069 (2)	C2—H2D	0.9600
Ni1—N3	2.079 (2)	C2—H2E	0.9600
Ni1—O4	2.092 (2)	C3—C4	1.509 (5)
Ni1—O1	2.098 (2)	C4—H4C	0.9600
Ni1—O3	2.138 (2)	C4—H4D	0.9600
Ni1—O2	2.164 (2)	C4—H4E	0.9600
N1—C9	1.351 (4)	C5—C6	1.400 (4)
N1—C5	1.351 (4)	C6—C7	1.358 (5)
N2—C5	1.346 (4)	C6—H6	0.9300
N2—H2A	0.8600	C7—C8	1.384 (5)
N2—H2B	0.8600	C7—H7	0.9300
N3—C10	1.339 (4)	C8—C9	1.354 (5)
N3—C14	1.353 (4)	C8—H8	0.9300
N4—C10	1.336 (4)	C9—H9	0.9300
N4—H4A	0.8600	C10—C11	1.403 (5)
N4—H4B	0.8600	C11—C12	1.345 (5)
O1—C1	1.264 (4)	C11—H11	0.9300
O2—C1	1.259 (4)	C12—C13	1.379 (6)
O3—C3	1.254 (4)	C12—H12	0.9300
O4—C3	1.264 (4)	C13—C14	1.354 (5)
C1—C2	1.497 (5)	C13—H13	0.9300
C2—H2C	0.9600	C14—H14	0.9300
N1—Ni1—N3	91.18 (10)	O3—C3—O4	119.7 (3)
N1—Ni1—O4	95.25 (9)	O3—C3—C4	120.6 (3)
N3—Ni1—O4	101.98 (9)	O4—C3—C4	119.7 (3)
N1—Ni1—O1	102.86 (9)	C3—C4—H4C	109.5
N3—Ni1—O1	95.05 (10)	C3—C4—H4D	109.5
O4—Ni1—O1	154.83 (9)	H4C—C4—H4D	109.5
N1—Ni1—O3	93.74 (9)	C3—C4—H4E	109.5
N3—Ni1—O3	163.58 (9)	H4C—C4—H4E	109.5
O4—Ni1—O3	61.99 (9)	H4D—C4—H4E	109.5
O1—Ni1—O3	99.10 (9)	N2—C5—N1	118.2 (3)
N1—Ni1—O2	164.38 (9)	N2—C5—C6	121.3 (3)
N3—Ni1—O2	89.06 (9)	N1—C5—C6	120.6 (3)
O4—Ni1—O2	99.98 (9)	C7—C6—C5	120.1 (3)
O1—Ni1—O2	61.58 (8)	C7—C6—H6	119.9
O3—Ni1—O2	90.37 (8)	C5—C6—H6	119.9
C9—N1—C5	117.5 (3)	C6—C7—C8	119.4 (3)
C9—N1—Ni1	115.6 (2)	C6—C7—H7	120.3
C5—N1—Ni1	126.8 (2)	C8—C7—H7	120.3
C5—N2—H2A	120.0	C9—C8—C7	118.1 (3)
C5—N2—H2B	120.0	C9—C8—H8	121.0
H2A—N2—H2B	120.0	C7—C8—H8	121.0
C10—N3—C14	117.2 (3)	N1—C9—C8	124.3 (3)
C10—N3—Ni1	126.4 (2)	N1—C9—H9	117.8

C14—N3—Ni1	116.2 (2)	C8—C9—H9	117.8
C10—N4—H4A	120.0	N4—C10—N3	118.4 (3)
C10—N4—H4B	120.0	N4—C10—C11	120.5 (3)
H4A—N4—H4B	120.0	N3—C10—C11	121.1 (3)
C1—O1—Ni1	90.71 (19)	C12—C11—C10	119.6 (3)
C1—O2—Ni1	87.88 (19)	C12—C11—H11	120.2
C3—O3—Ni1	88.18 (19)	C10—C11—H11	120.2
C3—O4—Ni1	90.01 (19)	C11—C12—C13	120.0 (4)
O2—C1—O1	119.7 (3)	C11—C12—H12	120.0
O2—C1—C2	121.1 (3)	C13—C12—H12	120.0
O1—C1—C2	119.2 (3)	C14—C13—C12	117.8 (4)
C1—C2—H2C	109.5	C14—C13—H13	121.1
C1—C2—H2D	109.5	C12—C13—H13	121.1
H2C—C2—H2D	109.5	C13—C14—N3	124.3 (3)
C1—C2—H2E	109.5	C13—C14—H14	117.9
H2C—C2—H2E	109.5	N3—C14—H14	117.9
H2D—C2—H2E	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O2^{i}	0.86	2.13	2.988 (3)	179
N2—H2*A*···O1	0.86	2.20	2.967 (3)	149
N4—H4*A*···O4	0.86	2.03	2.848 (4)	158

Symmetry code: (i) $x+1/2, y, -z+1/2$.