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trans-Bis[2-(4-ethyl-4,5-dihydro-1,3-oxazol-2-yl)phenolato-κ^2N,O^1]nickel(II)

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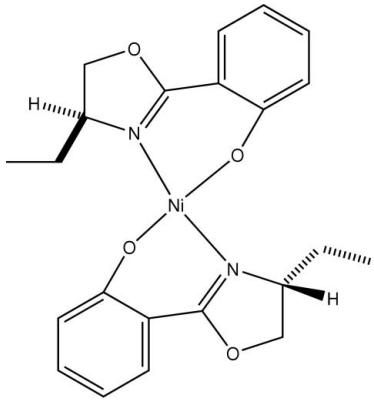
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.002 Å; R factor = 0.027; wR factor = 0.078; data-to-parameter ratio = 18.4.

In the title centrosymmetric compound, $[Ni(C_{11}H_{12}NO_2)_2]$, the Ni^{II} ion is located on an inversion centre in a square-planar coordination geometry, with Ni—N distances of 1.8856 (11) Å and Ni—O distances of 1.8484 (10) Å. The crystal packing is stabilized by π–π stacking, the centroid-to-centroid distance being 3.748 (1) Å. A C—H$\cdots\pi$ interaction is also observed in the crystal structure.

Related literature

For general background, see: Cozzi *et al.* (1995); Braunstein & Naud (2001); Kandasamy *et al.* (2004); Zhang *et al.* (2007). For synthesis, see: Serrano *et al.* (1995).



Experimental

Crystal data

$[Ni(C_{11}H_{12}NO_2)_2]$
M_r = 439.14
Monoclinic, $P2_1/c$
a = 6.8507 (8) Å
b = 14.2231 (16) Å
c = 10.4333 (11) Å
β = 94.978 (1)°
V = 1012.8 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.99 mm^{-1}
T = 273 (2) K
0.46 × 0.37 × 0.26 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.660, T_{max} = 0.785
9124 measured reflections
2464 independent reflections
2117 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.078
S = 1.04
2464 reflections
134 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

Cg is the centroid of the benzene ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C8—H8$B\cdots Cg^{i}$	0.97	2.77	3.552 (2)	138
C8—H8$A\cdots Cg^{ii}$	0.97	2.92	3.769 (2)	147

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

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2276).

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supplementary materials

Acta Cryst. (2007). E**63**, m2231 [**doi:10.1107/S1600536807035829**]

trans-Bis[2-(4-ethyl-4,5-dihydro-1,3-oxazol-2-yl)phenolato-κ^2N,O^1]nickel(II)

Y. Zhang, T.-F. Liu and W.-G. Xu

Comment

The chemistry of oxazoline-based ligands continues to be an area of interest due to its ability to form kinetically inert chiral metal complexes of potential for asymmetric synthesis. In particular, oxazoline has been used as a chiral auxiliary in catalytic alkene cyclopropanation, in palladium-catalyzed allylic coupling, and in Diels-Alder reactions. Several metal complexes bearing 2-(2'-hydroxyphenyl)oxazolines have been reported in the literature (Cozzi *et al.*, 1995; Braunstein *et al.*, 2001; Kandasamy *et al.*, 2004; Zhang *et al.*, 2007).

We report here the crystal structure of the title compound, a Ni(II) complex with chiral ligand, 2-(4-ethyl-4,5-dihydrooxazol −2-yl)phenol, as coordination ligand.

The title compound, contains one centrosymmetric tetra-coordinated nickel(II) complex (Fig 1). The nickel atom is coordinated by two 2-(4-ethyl- 4,5-dihydrooxazol-2-yl)-phenol anions, with N atom and phenolyl O atom as coordination atoms. Pairs of equivalent ligands lie *trans* to each other in a slightly distorted square geometry about the nickel(II) atom. The distances of Ni–O are 1.8484 (10) Å, and the distances of Ni–N are 1.8856 (11) Å. The angle O1–Ni1–N1 is 92.22 (5) °, and the angle O1–Ni1–N1^{i} is 87.78 (5) ° (symmetry code: (i) $-x+1, -y+1, -z+2$).

The aryl and oxazoline least-squares planes are linked by π-π stacking interactions with *Cg*-*Cg*^{ii} distances 3.7475 (10) Å (symmetry code: (ii) $2-x, 1-y, 2-z$). The C—H···*Cg* (aryl ring) interactions are observed with H8A^{iii}···*Cg* = 2.77 Å (symmetry code: (iii) $x, 1/2-y, -1/2+z$) and H(8B)^{i}···*Cg* = 2.920 Å (Spek, 2003).

Experimental

The racemic ligand, 2-(4-ethyl-4,5-dihydrooxazol-2-yl)phenol was prepared from 2-hydroxybenzonitrile and racemic 2-aminobutan-1-ol as literature reported (Serrano *et al.*, 1995).

A solution of 2-(4-ethyl-4,5-dihydrooxazol-2-yl)phenol (30.56 mg, 0.16 mmol) in methanol (4 ml) was added to a stirred solution of $Ni(NO_3)_2{\cdot}6H_2O$ (58.16 mg, 0.2 mmol) in methanol (2 ml). Crystals suitable for diffraction analysis were obtained after a few days.

Refinement

H atoms were positioned geometrically (aromatic C—H = 0.93 Å, aliphatic C—H = 0.96–0.98 Å) and were included in the refinement in the riding model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$ [$1.5U_{eq}(C)$ for methyl H].

Figures

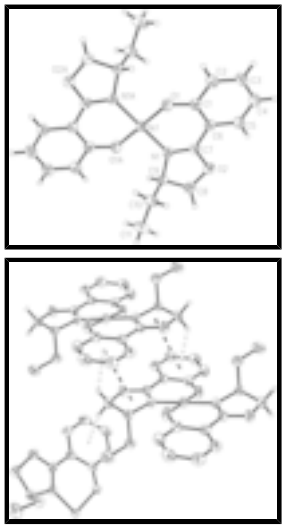

Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level (symmetry code: $-x + 1, -y + 1, -z + 2$).

trans-Bis[2-(4-ethyl-4,5-dihydro-1,3-oxazol-2-yl)phenolato-\ κ^2N,O^1]nickel(II)

Crystal data

$[Ni(C_{11}H_{12}NO_2)_2]$	$F_{000} = 460$
$M_r = 439.14$	$D_x = 1.440$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 4600 reflections
$a = 6.8507$ (8) Å	$\theta = 2.4$–27.9º
$b = 14.2231$ (16) Å	$\mu = 0.99$ mm^{-1}
$c = 10.4333$ (11) Å	$T = 273$ (2) K
$\beta = 94.978$ (1)º	Block, green
$V = 1012.8$ (2) Å^3	0.46 × 0.37 × 0.26 mm
$Z = 2$	

Data collection

Bruker SMART CCD area-detector diffractometer	2464 independent reflections
Radiation source: fine-focus sealed tube	2117 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.015$
Detector resolution: 0 pixels mm^{-1}	$\theta_{max} = 28.2$º
$T = 273(2)$ K	$\theta_{min} = 2.4$º
φ and ω scans	$h = -8 \rightarrow 9$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -18 \rightarrow 18$
$T_{min} = 0.660$, $T_{max} = 0.785$	$l = -13 \rightarrow 13$
9124 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.027$	H-atom parameters constrained
$wR(F^2) = 0.078$	$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 0.2109P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
2464 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
134 parameters	$\Delta\rho_{min} = -0.14$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.5000	0.5000	1.0000	0.04033 (10)
N1	0.69407 (17)	0.42879 (8)	0.92667 (11)	0.0416 (3)
O1	0.58717 (16)	0.46227 (9)	1.16463 (10)	0.0539 (3)
C7	0.8531 (2)	0.39736 (10)	0.98742 (14)	0.0421 (3)
O2	0.98132 (16)	0.35575 (8)	0.91413 (11)	0.0537 (3)
C1	0.7605 (2)	0.42966 (10)	1.20546 (14)	0.0448 (3)
C6	0.9035 (2)	0.39973 (10)	1.12425 (14)	0.0427 (3)
C9	0.6929 (2)	0.40152 (10)	0.78937 (13)	0.0435 (3)
H9	0.6695	0.4571	0.7346	0.052*
C2	0.8101 (3)	0.42306 (12)	1.33957 (15)	0.0570 (4)
H2	0.7180	0.4399	1.3958	0.068*
C10	0.5365 (3)	0.32755 (12)	0.75391 (16)	0.0576 (4)
H10A	0.4122	0.3486	0.7816	0.069*
H10B	0.5718	0.2694	0.7987	0.069*
C3	0.9921 (3)	0.39224 (13)	1.38856 (17)	0.0628 (4)
H3	1.0207	0.3887	1.4772	0.075*
C5	1.0889 (2)	0.36959 (11)	1.17710 (17)	0.0523 (4)
H5	1.1825	0.3516	1.1225	0.063*
C8	0.9026 (2)	0.36654 (13)	0.78168 (16)	0.0543 (4)
H8A	0.9034	0.3070	0.7365	0.065*
H8B	0.9786	0.4118	0.7374	0.065*
C4	1.1339 (3)	0.36634 (13)	1.30740 (18)	0.0619 (4)
H4	1.2575	0.3471	1.3413	0.074*
C11	0.5138 (3)	0.30932 (16)	0.60927 (18)	0.0801 (6)
H11A	0.6392	0.2939	0.5803	0.120*
H11B	0.4633	0.3647	0.5654	0.120*
H11C	0.4249	0.2579	0.5911	0.120*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.04070 (15)	0.04505 (16)	0.03585 (14)	0.00470 (10)	0.00680 (10)	0.00219 (9)
N1	0.0441 (6)	0.0435 (6)	0.0378 (6)	0.0015 (5)	0.0066 (5)	−0.0002 (5)
O1	0.0511 (6)	0.0718 (7)	0.0397 (5)	0.0146 (5)	0.0088 (4)	0.0078 (5)
C7	0.0431 (7)	0.0362 (7)	0.0479 (7)	−0.0004 (5)	0.0081 (6)	−0.0024 (5)
O2	0.0501 (6)	0.0588 (7)	0.0529 (6)	0.0121 (5)	0.0089 (5)	−0.0072 (5)
C1	0.0492 (8)	0.0422 (7)	0.0426 (7)	0.0006 (6)	0.0023 (6)	0.0046 (6)
C6	0.0452 (7)	0.0365 (7)	0.0460 (7)	−0.0003 (5)	0.0018 (6)	0.0016 (5)
C9	0.0505 (8)	0.0427 (7)	0.0381 (7)	−0.0017 (6)	0.0086 (6)	−0.0020 (5)
C2	0.0667 (10)	0.0601 (10)	0.0442 (8)	0.0050 (8)	0.0040 (7)	0.0061 (7)
C10	0.0638 (10)	0.0573 (9)	0.0518 (9)	−0.0153 (8)	0.0061 (7)	−0.0030 (7)
C3	0.0763 (12)	0.0607 (10)	0.0485 (9)	0.0015 (8)	−0.0120 (8)	0.0070 (7)
C5	0.0472 (8)	0.0469 (8)	0.0618 (10)	0.0034 (6)	−0.0007 (7)	−0.0008 (7)
C8	0.0557 (9)	0.0571 (9)	0.0512 (9)	0.0039 (7)	0.0118 (7)	−0.0107 (7)
C4	0.0585 (10)	0.0580 (10)	0.0657 (11)	0.0051 (8)	−0.0145 (8)	0.0044 (8)
C11	0.0972 (16)	0.0866 (14)	0.0549 (11)	−0.0296 (12)	−0.0031 (10)	−0.0124 (10)

Geometric parameters (Å, °)

Ni1—O1^{i}	1.8484 (10)	C2—C3	1.377 (2)
Ni1—O1	1.8484 (10)	C2—H2	0.9300
Ni1—N1	1.8856 (11)	C10—C11	1.526 (2)
Ni1—N1^{i}	1.8856 (11)	C10—H10A	0.9700
N1—C7	1.2917 (18)	C10—H10B	0.9700
N1—C9	1.4834 (17)	C3—C4	1.393 (3)
O1—C1	1.3113 (18)	C3—H3	0.9300
C7—O2	1.3503 (16)	C5—C4	1.369 (2)
C7—C6	1.440 (2)	C5—H5	0.9300
O2—C8	1.447 (2)	C8—H8A	0.9700
C1—C2	1.414 (2)	C8—H8B	0.9700
C1—C6	1.415 (2)	C4—H4	0.9300
C6—C5	1.407 (2)	C11—H11A	0.9600
C9—C10	1.524 (2)	C11—H11B	0.9600
C9—C8	1.529 (2)	C11—H11C	0.9600
C9—H9	0.9800		
O1^{i}—Ni1—O1	180.0	C1—C2—H2	119.3
O1^{i}—Ni1—N1	87.78 (5)	C9—C10—C11	111.49 (14)
O1—Ni1—N1	92.22 (5)	C9—C10—H10A	109.3
O1^{i}—Ni1—N1^{i}	92.22 (5)	C11—C10—H10A	109.3
O1—Ni1—N1^{i}	87.78 (5)	C9—C10—H10B	109.3
N1—Ni1—N1^{i}	180.000 (1)	C11—C10—H10B	109.3
C7—N1—C9	108.48 (12)	H10A—C10—H10B	108.0
C7—N1—Ni1	125.53 (10)	C2—C3—C4	121.02 (16)
C9—N1—Ni1	125.97 (9)	C2—C3—H3	119.5

C1—O1—Ni1	127.96 (9)	C4—C3—H3	119.5
N1—C7—O2	115.87 (13)	C4—C5—C6	121.17 (16)
N1—C7—C6	126.71 (13)	C4—C5—H5	119.4
O2—C7—C6	117.39 (13)	C6—C5—H5	119.4
C7—O2—C8	106.70 (11)	O2—C8—C9	104.97 (11)
O1—C1—C2	118.56 (14)	O2—C8—H8A	110.8
O1—C1—C6	124.49 (13)	C9—C8—H8A	110.8
C2—C1—C6	116.96 (14)	O2—C8—H8B	110.8
C5—C6—C1	120.34 (14)	C9—C8—H8B	110.8
C5—C6—C7	120.79 (14)	H8A—C8—H8B	108.8
C1—C6—C7	118.82 (13)	C5—C4—C3	119.07 (16)
N1—C9—C10	111.07 (12)	C5—C4—H4	120.5
N1—C9—C8	102.09 (12)	C3—C4—H4	120.5
C10—C9—C8	113.99 (14)	C10—C11—H11A	109.5
N1—C9—H9	109.8	C10—C11—H11B	109.5
C10—C9—H9	109.8	H11A—C11—H11B	109.5
C8—C9—H9	109.8	C10—C11—H11C	109.5
C3—C2—C1	121.36 (16)	H11A—C11—H11C	109.5
C3—C2—H2	119.3	H11B—C11—H11C	109.5
$O1^{i}$—Ni1—N1—C7	161.17 (13)	O2—C7—C6—C5	7.2 (2)
O1—Ni1—N1—C7	−18.83 (13)	N1—C7—C6—C1	7.4 (2)
$O1^{i}$—Ni1—N1—C9	−17.12 (11)	O2—C7—C6—C1	−170.52 (13)
O1—Ni1—N1—C9	162.88 (11)	C7—N1—C9—C10	110.60 (15)
N1—Ni1—O1—C1	21.57 (14)	Ni1—N1—C9—C10	−70.87 (15)
$N1^{i}$—Ni1—O1—C1	−158.43 (14)	C7—N1—C9—C8	−11.28 (15)
C9—N1—C7—O2	4.75 (17)	Ni1—N1—C9—C8	167.25 (10)
Ni1—N1—C7—O2	−173.78 (9)	O1—C1—C2—C3	−177.90 (16)
C9—N1—C7—C6	−173.18 (13)	C6—C1—C2—C3	2.4 (2)
Ni1—N1—C7—C6	8.3 (2)	N1—C9—C10—C11	171.80 (15)
N1—C7—O2—C8	4.60 (17)	C8—C9—C10—C11	−73.5 (2)
C6—C7—O2—C8	−177.26 (13)	C1—C2—C3—C4	−0.1 (3)
Ni1—O1—C1—C2	167.16 (12)	C1—C6—C5—C4	1.6 (2)
Ni1—O1—C1—C6	−13.2 (2)	C7—C6—C5—C4	−176.08 (15)
O1—C1—C6—C5	177.19 (15)	C7—O2—C8—C9	−11.42 (16)
C2—C1—C6—C5	−3.1 (2)	N1—C9—C8—O2	13.45 (15)
O1—C1—C6—C7	−5.1 (2)	C10—C9—C8—O2	−106.41 (14)
C2—C1—C6—C7	174.57 (14)	C6—C5—C4—C3	0.9 (3)
N1—C7—C6—C5	−174.93 (14)	C2—C3—C4—C5	−1.6 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8B···Cg^{ii}	0.97	2.77	3.552 (2)	138
C8—H8A···Cg^{iii}	0.97	2.92	3.769 (2)	147

Symmetry codes: (ii) −*x*+2, −*y*+1, −*z*+2; (iii) *x*, −*y*+1/2, *z*−1/2.

Fig. 1

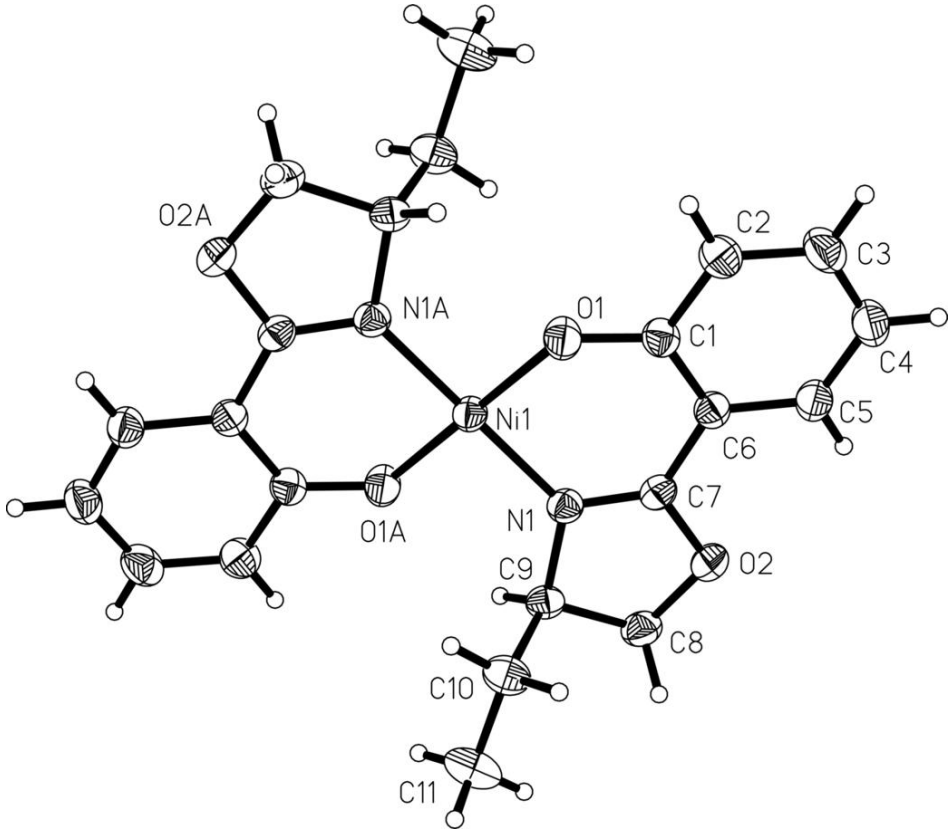

Fig. 2