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Bis[2-(3-cyanophenyliminomethyl)-phenolato]nickel(II)

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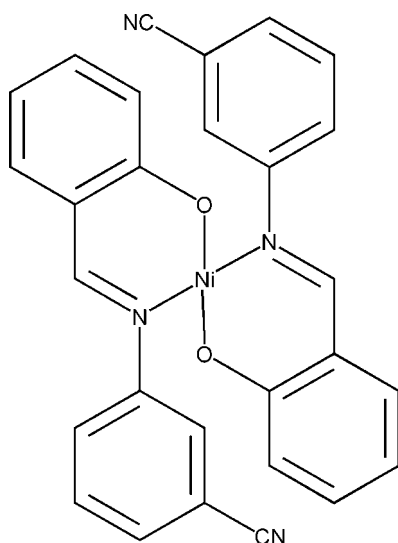
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
 R factor = 0.034; wR factor = 0.090; data-to-parameter ratio = 15.9.

In the title complex, $[\text{Ni}(\text{C}_{14}\text{H}_9\text{N}_2\text{O})_2]$, the Ni^{II} atom lies on an inversion center and is coordinated by the O atom and an N atom of two Schiff base 2-(3-cyanophenyliminomethyl)-phenolate ligands in a square-planar geometry. The dihedral angle between the cyanophenyl and phenolate rings is 47.62 (7)°.

Related literature

For related literature, see: Adams *et al.* (2004); Bian *et al.* (2004); Brückner *et al.* (2000); Harrop *et al.* (2003); Marganian *et al.* (1995); Akkurt *et al.* (2006); Peng *et al.* (2006).



Experimental

Crystal data

$[\text{Ni}(\text{C}_{14}\text{H}_9\text{N}_2\text{O})_2]$
 $M_r = 501.17$
Monoclinic, $P2_1/c$
 $a = 9.0294$ (18) Å
 $b = 8.0856$ (16) Å
 $c = 15.644$ (3) Å
 $\beta = 104.01$ (3)°

$V = 1108.1$ (4) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.91$ mm⁻¹
 $T = 293$ (2) K
 $0.25 \times 0.18 \times 0.18$ mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan
(*CrystalClear*; Rigaku, 2005)
 $T_{\text{min}} = 0.852$, $T_{\text{max}} = 1.00$
(expected range = 0.723–0.849)

11052 measured reflections
2540 independent reflections
2246 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.090$
 $S = 1.10$
2540 reflections

160 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.32$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.35$ e Å⁻³

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-III* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2310).

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supporting information

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Bis[2-(3-cyanophenyliminomethyl)phenolato]nickel(II)**Xing-Xuan Gong, Rong Xia and Hai-Jun Xu****S1. Comment**

Schiff bases have been used extensively as ligands in the field of coordination chemistry. These complexes play an important role in the development of pharmacological and catalytic properties (Harrop *et al.*, 2003; Brückner *et al.*, 2000). Nickel(II) compounds with Schiff bases have received much attention in recent years (Marganian *et al.*, 1995). Here we report the molecular and crystal structure of nickel (II) complex with a Schiff base ligand.

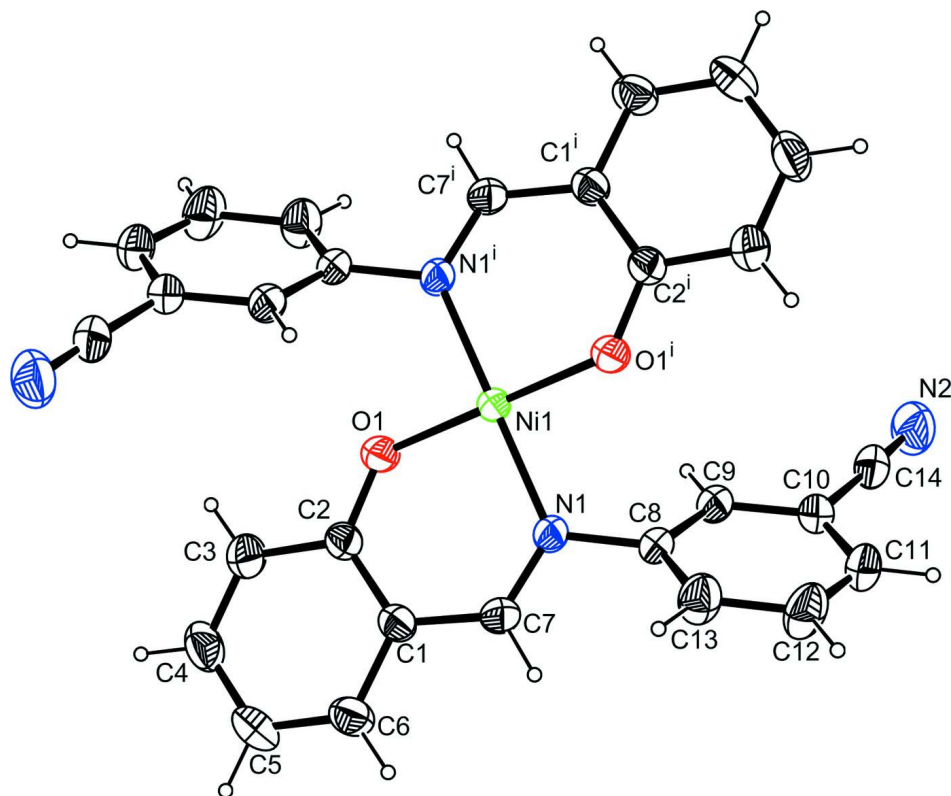
The Ni^{II} atom in (I) lies on an inversion center and is coordinated by the two imine N and two phenolate O atoms of the two Schiff base ligands in a square-planar geometry (Fig.1). The dihedral angle between the cyanophenyl and phenyl rings is 47.62 (7)°. The Ni—O and Ni—N bond lengths agree with the values reported for related complexes (Peng, *et al.*, (2006); Adams *et al.*, 2004; Bian *et al.*, 2004).

S2. Experimental

2-(3-cyanophenyliminomethyl)phenol was prepared according to the literature (Akkurt *et al.*, 2006). NiCl₂·6H₂O (23.7 mg, 0.1 mmol) in methanol (5 ml) was added to the solution of 2-(3-cyanophenyliminomethyl)phenol (22.2 mg, 0.1 mmol) in the methanol (5 ml), the pH was then adjusted to 8–9 and the mixture was stirred for 4 h. The filtrate was kept at room temperature for about two weeks, and blue block shaped crystals of (I) suitable for X-ray single-crystal analyses were obtained.

S3. Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

Molecular view of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii. [Symmetry code: (i) $1 - x, 1 - y, 1 - z$]

Bis[2-(3-cyanophenyliminomethyl)phenolato]nickel(II)

Crystal data

$[\text{Ni}(\text{C}_{14}\text{H}_9\text{N}_2\text{O})_2]$

$M_r = 501.17$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 9.0294\ (18)\ \text{\AA}$

$b = 8.0856\ (16)\ \text{\AA}$

$c = 15.644\ (3)\ \text{\AA}$

$\beta = 104.01\ (3)^\circ$

$V = 1108.1\ (4)\ \text{\AA}^3$

$Z = 2$

$F(000) = 516$

$D_x = 1.502\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 10336 reflections

$\theta = 3.1\text{--}27.4^\circ$

$\mu = 0.91\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, blue

$0.25 \times 0.18 \times 0.18\ \text{mm}$

Data collection

Rigaku Mercury2

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: $13.6612\ \text{pixels mm}^{-1}$

ω scans

Absorption correction: multi-scan

(*CrystalClear*; Rigaku, 2005)

$T_{\min} = 0.852, T_{\max} = 1.00$

11052 measured reflections

2540 independent reflections

2246 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 27.5^\circ, \theta_{\min} = 3.1^\circ$

$h = -11 \rightarrow 11$

$k = -10 \rightarrow 10$

$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.090$
 $S = 1.10$
 2540 reflections
 160 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
 $w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.5389P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.5000	0.5000	0.5000	0.02514 (11)
O1	0.66446 (15)	0.36034 (17)	0.52053 (9)	0.0349 (3)
N1	0.41172 (16)	0.38940 (18)	0.58415 (10)	0.0266 (3)
C9	0.3215 (2)	0.6210 (2)	0.65595 (12)	0.0302 (4)
H9	0.4159	0.6714	0.6613	0.036*
C14	0.2380 (2)	0.8635 (3)	0.72431 (15)	0.0401 (5)
C1	0.5678 (2)	0.1429 (2)	0.59534 (12)	0.0283 (4)
C10	0.2087 (2)	0.6997 (2)	0.68770 (12)	0.0318 (4)
C8	0.2929 (2)	0.4678 (2)	0.61652 (12)	0.0274 (4)
C11	0.0679 (2)	0.6238 (3)	0.68133 (14)	0.0395 (5)
H11	-0.0069	0.6759	0.7034	0.047*
C3	0.7949 (2)	0.1080 (3)	0.54150 (14)	0.0383 (5)
H3	0.8663	0.1492	0.5130	0.046*
C2	0.6729 (2)	0.2098 (2)	0.55130 (11)	0.0286 (4)
C13	0.1528 (2)	0.3938 (3)	0.60894 (15)	0.0404 (5)
H13	0.1329	0.2914	0.5815	0.048*
C7	0.4475 (2)	0.2409 (2)	0.61321 (12)	0.0297 (4)
H7	0.3897	0.1942	0.6488	0.036*
C5	0.7059 (3)	-0.1167 (3)	0.61697 (15)	0.0432 (5)
H5	0.7169	-0.2245	0.6382	0.052*
N2	0.2618 (3)	0.9944 (2)	0.75059 (17)	0.0570 (6)
C12	0.0409 (2)	0.4716 (3)	0.64212 (17)	0.0461 (6)
H12	-0.0527	0.4199	0.6377	0.055*
C4	0.8094 (3)	-0.0502 (3)	0.57343 (15)	0.0430 (5)
H4	0.8904	-0.1149	0.5658	0.052*

C6	0.5875 (3)	-0.0197 (2)	0.62794 (14)	0.0366 (4)
H6	0.5184	-0.0625	0.6577	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02507 (18)	0.02233 (17)	0.03093 (19)	0.00178 (12)	0.01241 (13)	0.00362 (12)
O1	0.0309 (7)	0.0327 (7)	0.0458 (8)	0.0070 (6)	0.0182 (6)	0.0125 (6)
N1	0.0262 (7)	0.0245 (7)	0.0319 (7)	-0.0009 (6)	0.0124 (6)	-0.0003 (6)
C9	0.0294 (9)	0.0286 (9)	0.0360 (9)	-0.0003 (7)	0.0146 (8)	0.0016 (7)
C14	0.0431 (12)	0.0363 (12)	0.0480 (12)	0.0067 (9)	0.0247 (10)	0.0000 (9)
C1	0.0305 (9)	0.0236 (9)	0.0309 (9)	0.0009 (7)	0.0075 (7)	0.0007 (7)
C10	0.0348 (10)	0.0305 (9)	0.0332 (9)	0.0058 (8)	0.0140 (8)	0.0022 (8)
C8	0.0282 (9)	0.0272 (9)	0.0300 (9)	0.0015 (7)	0.0133 (7)	0.0023 (7)
C11	0.0327 (10)	0.0449 (12)	0.0461 (11)	0.0093 (9)	0.0194 (9)	0.0032 (9)
C3	0.0335 (10)	0.0412 (11)	0.0425 (11)	0.0088 (9)	0.0134 (9)	0.0043 (9)
C2	0.0294 (9)	0.0282 (9)	0.0278 (9)	0.0036 (7)	0.0064 (7)	0.0020 (7)
C13	0.0348 (10)	0.0378 (11)	0.0521 (12)	-0.0068 (9)	0.0174 (9)	-0.0090 (9)
C7	0.0315 (9)	0.0263 (9)	0.0335 (9)	-0.0034 (7)	0.0122 (7)	0.0022 (7)
C5	0.0491 (13)	0.0250 (10)	0.0528 (13)	0.0073 (9)	0.0070 (10)	0.0048 (9)
N2	0.0685 (15)	0.0369 (11)	0.0762 (15)	-0.0001 (9)	0.0379 (12)	-0.0109 (10)
C12	0.0294 (10)	0.0550 (14)	0.0592 (14)	-0.0081 (9)	0.0212 (10)	-0.0084 (11)
C4	0.0407 (12)	0.0397 (11)	0.0476 (12)	0.0173 (9)	0.0085 (9)	0.0018 (10)
C6	0.0398 (11)	0.0266 (10)	0.0437 (11)	-0.0013 (8)	0.0104 (9)	0.0045 (8)

Geometric parameters (Å, °)

Ni1—O1 ⁱ	1.8310 (14)	C8—C13	1.378 (3)
Ni1—O1	1.8310 (14)	C11—C12	1.371 (3)
Ni1—N1 ⁱ	1.9174 (15)	C11—H11	0.9300
Ni1—N1	1.9174 (15)	C3—C4	1.368 (3)
O1—C2	1.304 (2)	C3—C2	1.413 (3)
N1—C7	1.297 (2)	C3—H3	0.9300
N1—C8	1.440 (2)	C13—C12	1.393 (3)
C9—C8	1.380 (3)	C13—H13	0.9300
C9—C10	1.391 (3)	C7—H7	0.9300
C9—H9	0.9300	C5—C6	1.370 (3)
C14—N2	1.137 (3)	C5—C4	1.390 (3)
C14—C10	1.442 (3)	C5—H5	0.9300
C1—C6	1.406 (2)	C12—H12	0.9300
C1—C2	1.409 (3)	C4—H4	0.9300
C1—C7	1.426 (3)	C6—H6	0.9300
C10—C11	1.393 (3)		
O1 ⁱ —Ni1—O1	180.000 (1)	C10—C11—H11	120.4
O1 ⁱ —Ni1—N1 ⁱ	92.65 (6)	C4—C3—C2	120.89 (19)
O1—Ni1—N1 ⁱ	87.35 (6)	C4—C3—H3	119.6
O1 ⁱ —Ni1—N1	87.35 (6)	C2—C3—H3	119.6

O1—Ni1—N1	92.65 (6)	O1—C2—C1	123.56 (17)
N1 ⁱ —Ni1—N1	180.000 (1)	O1—C2—C3	118.75 (17)
C2—O1—Ni1	127.78 (12)	C1—C2—C3	117.68 (17)
C7—N1—C8	115.34 (15)	C8—C13—C12	120.4 (2)
C7—N1—Ni1	124.25 (13)	C8—C13—H13	119.8
C8—N1—Ni1	120.35 (12)	C12—C13—H13	119.8
C8—C9—C10	119.75 (18)	N1—C7—C1	125.63 (17)
C8—C9—H9	120.1	N1—C7—H7	117.2
C10—C9—H9	120.1	C1—C7—H7	117.2
N2—C14—C10	177.8 (2)	C6—C5—C4	118.5 (2)
C6—C1—C2	119.71 (18)	C6—C5—H5	120.7
C6—C1—C7	118.93 (18)	C4—C5—H5	120.7
C2—C1—C7	121.21 (16)	C11—C12—C13	120.4 (2)
C9—C10—C11	120.51 (18)	C11—C12—H12	119.8
C9—C10—C14	118.81 (18)	C13—C12—H12	119.8
C11—C10—C14	120.64 (18)	C3—C4—C5	121.6 (2)
C13—C8—C9	119.78 (17)	C3—C4—H4	119.2
C13—C8—N1	121.69 (17)	C5—C4—H4	119.2
C9—C8—N1	118.53 (16)	C5—C6—C1	121.6 (2)
C12—C11—C10	119.20 (19)	C5—C6—H6	119.2
C12—C11—H11	120.4	C1—C6—H6	119.2

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