C10*A*	0.1969 (4)	0.2853 (2)	0.13686 (13)	0.0508 (6)
C11*A*	0.2325 (3)	0.3410 (2)	0.08395 (13)	0.0474 (5)
C12*A*	0.2670 (4)	0.2979 (2)	0.00840 (13)	0.0500 (6)
C13*A*	0.2684 (3)	0.2058 (2)	−0.01183 (13)	0.0474 (5)
C14*A*	0.2343 (3)	0.1507 (2)	0.04111 (12)	0.0450 (5)
C15*A*	0.2346 (3)	0.0532 (2)	0.02316 (14)	0.0484 (5)
C16*A*	0.2683 (4)	0.0018 (2)	−0.04342 (14)	0.0502 (6)
C17*A*	0.2714 (3)	−0.0947 (2)	−0.05712 (12)	0.0438 (5)
C18*A*	0.3172 (4)	−0.1407 (2)	−0.12901 (14)	0.0519 (6)
C19*A*	0.3195 (4)	−0.2309 (2)	−0.14436 (14)	0.0528 (6)
C20*A*	0.2346 (4)	−0.2376 (2)	−0.02067 (14)	0.0543 (6)
C21*A*	0.2298 (4)	−0.1480 (2)	−0.00238 (14)	0.0521 (6)
C22*A*	0.2578 (7)	0.4893 (3)	0.0511 (2)	0.0779 (10)
C23*A*	0.2434 (7)	0.4809 (3)	0.1864 (2)	0.0784 (10)
C24*A*	0.2762 (6)	−0.3789 (2)	−0.1088 (2)	0.0718 (8)
N5*B*	0.1694 (3)	0.40742 (13)	0.61611 (11)	0.0531 (5)
N6*B*	0.3070 (3)	−0.29303 (13)	0.43527 (11)	0.0456 (4)
C9*B*	0.2612 (3)	0.1888 (2)	0.64186 (12)	0.0438 (5)
C10*B*	0.2402 (3)	0.2780 (2)	0.65959 (12)	0.0453 (5)
C11*B*	0.1963 (3)	0.31897 (15)	0.60023 (12)	0.0405 (5)
C12*B*	0.1766 (3)	0.2652 (2)	0.52332 (12)	0.0419 (5)
C13*B*	0.1969 (3)	0.1766 (2)	0.50690 (12)	0.0422 (5)
C14*B*	0.2389 (3)	0.13488 (15)	0.56573 (12)	0.0394 (5)
C15*B*	0.2602 (3)	0.0403 (2)	0.55064 (12)	0.0416 (5)
C16*B*	0.2395 (3)	−0.0195 (2)	0.48172 (13)	0.0421 (5)
C17*B*	0.2638 (3)	−0.11269 (15)	0.46800 (12)	0.0391 (4)
C18*B*	0.2369 (3)	−0.1682 (2)	0.39248 (12)	0.0466 (5)
C19*B*	0.2591 (4)	−0.2563 (2)	0.37714 (14)	0.0509 (6)
C20*B*	0.3333 (3)	−0.2416 (2)	0.50888 (14)	0.0507 (6)
C21*B*	0.3116 (4)	−0.1540 (2)	0.52662 (13)	0.0487 (6)
C22*B*	0.1659 (5)	0.4539 (2)	0.5536 (2)	0.0570 (7)
C23*B*	0.2061 (6)	0.4648 (2)	0.6948 (2)	0.0652 (8)
C24*B*	0.3320 (5)	−0.3885 (2)	0.4166 (2)	0.0620 (7)

Table 2. *Selected geometric parameters (Å, °)*

Anion			
Ni—S4	2.1658 (7)	N2—C4	1.143 (3)
Ni—S1	2.1766 (7)	N3—C7	1.146 (3)
Ni—S2	2.1787 (7)	N4—C8	1.150 (3)
Ni—S3	2.1805 (7)	C1—C2	1.355 (3)
S1—C1	1.732 (2)	C1—C3	1.429 (3)
S2—C2	1.735 (2)	C2—C4	1.424 (3)
S3—C5	1.730 (2)	C5—C6	1.357 (3)
S4—C6	1.728 (2)	C5—C7	1.428 (3)
N1—C3	1.140 (3)	C6—C8	1.432 (3)
S4—Ni—S1	177.48 (2)	S4—Ni—S3	92.22 (3)
S4—Ni—S2	86.92 (3)	S1—Ni—S3	88.64 (3)
S1—Ni—S2	92.31 (3)	S2—Ni—S3	177.64 (2)

	Cation *A*	Cation *B*
N5—C11	1.370 (3)	1.377 (3)
N5—C22	1.440 (4)	1.450 (3)
N5—C23	1.442 (4)	1.450 (3)
N6—C19	1.344 (3)	1.352 (3)
N6—C20	1.351 (3)	1.343 (3)
N6—C24	1.473 (4)	1.477 (3)
C9—C10	1.369 (3)	1.374 (3)
C9—C14	1.393 (3)	1.394 (3)
C10—C11	1.405 (3)	1.404 (3)
C11—C12	1.407 (3)	1.406 (3)
C12—C13	1.376 (3)	1.363 (3)
C13—C14	1.399 (3)	1.399 (3)
C14—C15	1.455 (3)	1.454 (3)
C15—C16	1.322 (3)	1.329 (3)
C16—C17	1.447 (3)	1.443 (3)
C17—C18	1.398 (3)	1.393 (3)
C17—C21	1.411 (3)	1.405 (3)
C18—C19	1.349 (4)	1.359 (3)
C20—C21	1.346 (4)	1.351 (3)

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990) by direct methods. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93*. Geometrical calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1233). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Clemenson, P. I. (1990). *Coord. Chem. Rev.* **106**, 171–203.
Manoharan, P. T., Noordik, J. H., de Boer, E. & Keijzers, C. P. (1981). *J. Chem. Phys.* **74**, 1980–1989.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Shan, B.-Z., Zhang, X.-M., You, X.-Z., Fun, H.-K. & Sivakumar, K. (1995). *Acta Cryst.* **C52**. In the press.
Sheldrick, G. M. (1990). *SHELXTL/PC. Structure Determination Software Programs.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Siemens (1994). *XSCANS. X-ray Single Crystal Analysis System.* Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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Bis(*N*-benzyl-2-hydroxy-1-naphthaldiminato)nickel(II)

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Abstract

The crystal structure of the title compound, bis-[1-(benzyliminomethyl)-2-naphtholato-*N*,*O*]nickel(II), $[Ni(C_{18}H_{14}NO)_2]$, has been determined. Two bidentate Schiff base ligands coordinate to the Ni atom in a square-planar arrangement.

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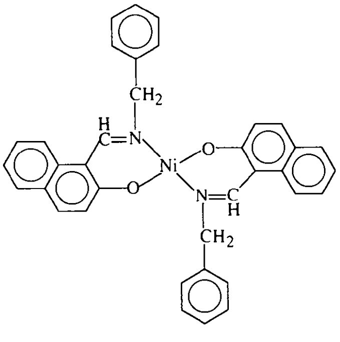
Comment

Schiff base metal complexes have been used extensively in the field of coordination chemistry, *e.g.* copper(II) and nickel(II) complexes. The electron delocalization to produce resonance structures of square-planar nickel(II) and copper(II) metal complexes with ligands derived from salicylaldehyde and naphthaldehyde has been examined previously (Fernández-G. *et al.*, 1987, and references therein). Maggio, Pizzino & Romano (1974) reported that copper(II) and nickel(II) ions react with tridentate anionic Schiff bases to form dimerized square-planar complexes. The same authors also reported that *N*-(2-hydroxyphenyl)salicylaldimine reacts with the nickel(II) ion to give a pseudo-octahedral complex by polymerization. The present paper reports the structure of the Schiff base nickel complex (I).



(I)

The Ni atom is surrounded by the N and O atoms in a square-planar arrangement. The two symmetry-equivalent bidentate ligands are bonded by the O and N atoms to the Ni atom, with distances Ni—O(1) and Ni—N(1) of 1.805 (3) and 1.915 (4) Å, respectively. These distances are in good agreement with those reported in similar four-coordinate square-planar nickel(II) complexes, as shown in Table 3. The O(1)—Ni—N(1) and O(1)—Ni—N(1*A*) bond angles of 91.6 (2) and 88.4 (2)°, respectively, show that the coordination sphere is square-planar. The dihedral angle between the chelate ring and the naphthaldimine group is 6.4 (2)°. This has been described previously as a stepped conformation (Fernández-G., Rosalez-Hoz, Toscano & Tapia, 1986) and has also been observed in other nickel complexes (Fernández-G. *et al.*, 1986, and references therein). Torsion angles in the Ni—O(1)—C(1)—C(10)—C(11)—N(1) chelate ring [clockwise from Ni—O(1)—C(1)—C(10) to N(1)—Ni—O(1)—C(1) −7.2 (7), −6.5 (8), 7.0 (8), 5.4 (8), −13.3 (4), 14.6 (4)°] reveal a very slight ring pucker. A search of the Cambridge Structural Database for octahedrally coordinated $\mathrm{Ni^{II}}$ ions resulted in 104 compounds with Ni—O and Ni—N bonds. The average Ni—O and Ni—N distances are 2.084 (6) and 2.110 (7) Å, respectively, which are significantly longer than those of square-planar coordination.

The bond length N(1)—C(11) is typical for a C=N double bond and the sum of the bond angles at the N(1) atom [360.0 (4)°] reflects the sp^2 hybridization.

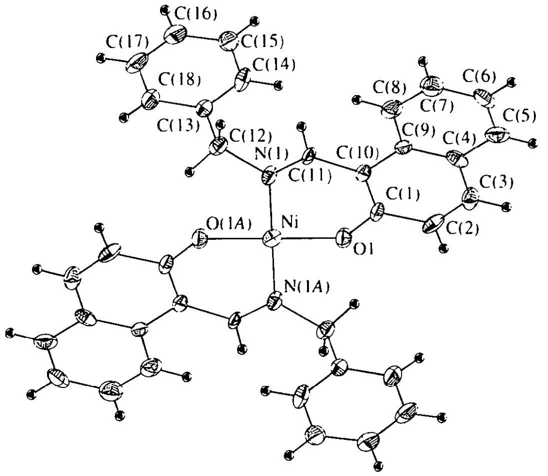


Fig. 1. Molecular structure of the title compound (*ORTEP*II; Johnson, 1976). Displacement ellipsoids are plotted at the 50% probability level.

Experimental

The title compound was obtained from the condensation of *N*-benzyl-2-hydroxynaphthaldimine and $(\mathrm{CH_3COO})_2\mathrm{Ni.4H_2O}$, prepared by standard procedures (Vogel, 1966).

Crystal data

$[\mathrm{Ni(C_{18}H_{14}NO)_2}]$
$M_r = 578.7$
Monoclinic
$C2/c$
$a = 20.756\ (1)$ Å
$b = 6.160\ (1)$ Å
$c = 21.475\ (1)$ Å
$\beta = 97.32\ (1)^\circ$
$V = 2723.2\ (5)$ Å^3
$Z = 4$
$D_x = 1.41$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 2–9^\circ$
$\mu = 0.696$ mm^{-1}
$T = 293\ (2)$ K
Prismatic
0.21 × 0.08 × 0.06 mm
Dark red

Data collection

CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction:
ψ scan (Kopfman & Huber, 1968; North, Phillips & Mathews, 1968)
$T_{\min} = 0.81$, $T_{\max} = 0.99$
1607 measured reflections
1178 independent reflections

689 observed reflections
$[I > 2\sigma(I)]$
$R_{\rm int} = 0.03$
$\theta_{\max} = 21.94^\circ$
$h = 0 \rightarrow 21$
$k = 0 \rightarrow 6$
$l = -22 \rightarrow 22$
3 standard reflections
frequency: 120 min
intensity decay: 4.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.075$
$S = 1.18$
689 reflections
187 parameters
H atoms riding with fixed $U_{\rm iso} = 1.6 \times U_{\rm eq}$ of the carrier atom
$w = 1/[\sigma^2(F_o^2) + (0.0267P)^2]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{\rm max} = 0.04$
$\Delta\rho_{\rm max} = 0.2$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.2$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Ni	3/4	1/4	1/2	0.037 (1)
O(1)	0.7393 (2)	0.5018 (6)	0.4570 (1)	0.043 (1)
N(1)	0.8229 (2)	0.1724 (6)	0.4583 (2)	0.033 (1)
C(1)	0.7633 (3)	0.5653 (9)	0.4069 (2)	0.033 (2)
C(2)	0.7409 (2)	0.7719 (11)	0.3804 (2)	0.042 (2)
C(3)	0.7633 (3)	0.8508 (9)	0.3289 (2)	0.044 (2)
C(4)	0.8100 (2)	0.7378 (11)	0.2982 (2)	0.038 (2)
C(5)	0.8305 (2)	0.8226 (9)	0.2432 (3)	0.050 (2)
C(6)	0.8728 (3)	0.7106 (11)	0.2119 (2)	0.050 (2)
C(7)	0.8952 (3)	0.5103 (10)	0.2340 (3)	0.054 (2)
C(8)	0.8760 (3)	0.4257 (10)	0.2880 (2)	0.047 (2)
C(9)	0.8320 (2)	0.5368 (9)	0.3222 (2)	0.031 (2)
C(10)	0.8098 (2)	0.4536 (9)	0.3784 (2)	0.032 (2)
C(11)	0.8383 (2)	0.2665 (11)	0.4094 (2)	0.034 (2)
C(12)	0.8654 (3)	−0.0117 (9)	0.4817 (2)	0.038 (2)
C(13)	0.9148 (2)	0.0479 (9)	0.5372 (2)	0.031 (2)
C(14)	0.9447 (3)	0.2474 (12)	0.5425 (2)	0.046 (2)
C(15)	0.9950 (3)	0.2948 (10)	0.5926 (3)	0.049 (2)
C(16)	1.0119 (3)	0.1360 (11)	0.6365 (3)	0.061 (2)
C(17)	0.9823 (3)	−0.0612 (12)	0.6320 (3)	0.059 (2)
C(18)	0.9346 (3)	−0.1097 (10)	0.5820 (3)	0.048 (2)

Table 2. *Selected geometric parameters (Å, °)*

Ni—O(1)	1.805 (3)	C(6)—C(7)	1.381 (8)
Ni—N(1)	1.915 (4)	C(7)—C(8)	1.376 (7)
O(1)—C(1)	1.301 (6)	C(8)—C(9)	1.419 (7)
N(1)—C(11)	1.275 (6)	C(9)—C(10)	1.441 (7)
N(1)—C(12)	1.484 (6)	C(10)—C(11)	1.422 (7)
C(1)—C(10)	1.388 (7)	C(12)—C(13)	1.516 (6)
C(1)—C(2)	1.446 (8)	C(13)—C(18)	1.392 (7)
C(2)—C(3)	1.343 (7)	C(13)—C(14)	1.374 (8)
C(3)—C(4)	1.423 (7)	C(14)—C(15)	1.432 (7)
C(4)—C(5)	1.405 (7)	C(15)—C(16)	1.373 (8)
C(4)—C(9)	1.395 (8)	C(16)—C(17)	1.359 (9)
C(5)—C(6)	1.360 (7)	C(17)—C(18)	1.397 (7)
O(1)—Ni—N(1)	91.6 (2)	O(1)—C(1)—C(2)	116.9 (5)
O(1)—Ni—O(1$A^{\rm i}$)	180.0 (2)	C(2)—C(1)—C(10)	118.0 (5)
O(1)—Ni—N(1$A^{\rm i}$)	88.4 (2)	C(4)—C(9)—C(8)	116.0 (5)
Ni—O(1)—C(1)	130.3 (3)	C(1)—C(10)—C(11)	117.9 (4)
C(11)—N(1)—C(12)	115.2 (4)	N(1)—C(11)—C(10)	128.3 (5)
Ni—N(1)—C(11)	124.6 (4)	N(1)—C(12)—C(13)	113.3 (4)
Ni—N(1)—C(12)	120.2 (3)	C(12)—C(13)—C(18)	119.0 (5)
O(1)—C(1)—C(10)	125.1 (5)		

Symmetry code: (i) $\frac{3}{2} - x, \frac{1}{2} - y, 1 - z$.

Table 3. *Bond lengths (Å) of some nickel(II) complexes*

Complex	Ni—N(1)	Ni—N(2)	Ni—O	C—O	C═N
(*a*)	1.848 (4)	1.947 (4)	1.817 (1)	1.315 (2)	1.292 (5)
(*b*)	1.849 (3)	1.955 (4)	1.831 (3)	1.322 (5)	1.303 (5)
(*c*)	1.916 (4)	–	1.829 (2)	1.321 (3)	1.300 (2)
(*d*)	1.912 (3)	–	1.832 (2)	1.305 (4)	1.297 (4)
(*e*)	1.941 (3)	–	1.828 (3)	1.332 (5)	1.279 (6)
(*f*)	1.854 (4)	1.970 (4)	1.828 (4)	1.335 (8)	1.263 (5)
(*g*)	1.898 (4)	1.936 (5)	1.848 (8)	1.322 (2)	1.285 (4)
(*h*)	1.915 (4)	–	1.805 (3)	1.301 (6)	1.275 (6)

Complex: (*a*) {1-[(2-Hydroxyphenyl)iminomethyl]naphthalen-2-olato-*O*,*O'*,*N*}piperidinenickel(II) (Elerman, Paulus & Fuess, 1991); (*b*) (diethylamino){1-[(2-hydroxyphenyl)iminomethyl]naphthalen-2-olato-*O*,*O'*,*N*}nickel(II) (Elerman, Fuess & Paulus, 1992); (*c*) bis{*N*-[(1,3-dioxolan-2-yl)methyl]salicylaldiminato}nickel(II) (Fernández-G. *et al.*, 1987); (*d*) bis{*N*-[(1,3-dioxolan-2-yl)methyl]-2-hydroxy-1-naphthaldiminato}nickel(II) (Fernández-G. *et al.*, 1987); (*e*) bis[*N*-(1,3-dioxolan-2-yl)-3-hydroxy-1-naphthaldiminato]nickel(II) (Fernández-G. *et al.*, 1987); (*f*) [*N*-(2-hydroxyphenyl)salicylaldiminato]amminenickel(II) (Elerman, Svoboda & Fuess, 1992); (*g*) [*N*-(2-hydroxyphenyl)salicylaldiminato](diethylamine)nickel(II) (Elerman, Paulus & Svoboda, 1992); (*h*) bis(*N*-benzyl-2-hydroxy-1-naphthaldiminato)nickel(II) (present work).

The structure was solved by direct phase determination. All non-H atoms were refined anisotropically by full-matrix least-squares methods. All atoms except the Ni atom, which is at a special position, were refined without any constraints. Values of distances and angles in the rings show no significant differences from those of an ideal benzene ring.

Data collection: *SDP-Plus* (Frenz, 1985). Cell refinement: *SDP-Plus*. Data reduction: *SDP-Plus*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

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Lists of structure factors, torsion angles, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1135). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Elerman, Y., Fuess, H. & Paulus, H. (1992). *Acta Cryst.* C**48**, 40–42.
Elerman, Y., Paulus, H. & Fuess, H. (1991). *Acta Cryst.* C**47**, 70–72.
Elerman, Y., Paulus, H. & Svoboda I. (1992). *Z. Kristallogr.* **198**, 132–134.
Elerman, Y., Svoboda, I. & Fuess, H. (1992). *Z. Kristallogr.* **198**, 127–128.
Fernández-G., J. M., Rosalez-Hoz, M. J., Rubiyo-Arroyo, M. F., Salcedo, R., Toscano, R. A. & Vela, A. (1987). *Inorg. Chem.* **26**, 349–357.
Fernández-G., J. M., Rosalez-Hoz, M. J., Toscano, R. A. & Tapia, T. R. G. (1986). *Acta Cryst.* C**42**, 1313–1316.
Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf–Nonius, Delft, The Netherlands.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Kopfman, G. & Huber, R. (1968). *Acta Cryst.* A**24**, 348–351.
Maggio, F., Pizzino, T. & Romano, V. (1974). *Inorg. Nucl. Chem. Lett.* **10**, 1005–1008.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Vogel, A. I. (1966). *Practical Organic Chemistry*, p. 653. London: Longmans.