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Structure of (Diethylamino){1-[(2-hydroxyphenyl)iminomethyl]naphthalen-2-olato-*O*,*O'*,*N*}nickel(II)

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(*Received* 2 *October* 1990; *accepted* 12 *April* 1991)

Abstract. $[\mathrm{Ni(C_4H_{11}N)(C_{17}H_{11}NO_2)}]$, $M_r = 393.11$, monoclinic, $P2_1/n$, $a = 12.233$ (4), $b = 8.846$ (3), $c = 17.155$ (6) Å, $\beta = 96.04$ (1)°, $V = 1846.2$ Å^3, $Z = 4$, $D_x = 1.414$ g cm^{-3}, $\lambda(\mathrm{Mo}K\alpha) = 0.71069$ Å, $\mu = 10.41$ cm^{-1}, $F(000) = 824$, $T = 298$ K, final $R = 0.035$, $wR = 0.030$ for 2181 unique reflections with $F > 2\sigma(F)$. The coordination of the Ni atom is square planar with Ni—O distances 1.820 (3) and 1.842 (3) Å, Ni—N distances 1.849 (3) and 1.955 (4) Å; the angles between the neighbouring ligands are in the range 86.8 to 94.8 (1)°. The diethylamino ligand is fully extended perpendicular to the Ni coordination plane.

Introduction. Metal derivatives of Schiff bases have been extensively studied and the copper(II) and nickel(II) complexes play a major role in both synthesis and structural research. Copper(II) and nickel(II) ions react with tridentate anionic Schiff bases and form dimer complexes with square-planar configurations as described by Maggio, Pizzino & Romano (1974). They report that *N*-(2-hydroxyphenyl)salicylaldimine reacts with nickel(II) ions giving a pseudo-octahedral complex by polymerization. We have investigated the reaction of nickel(II) with the monodentate ligand diethylamine and the tridentate ligand *N*-(2-hydroxyphenyl)-2-hydroxy-1-naphthaldimine; the structure of the reaction product is reported in this paper.

Experimental. Suitable crystals were obtained directly from the synthesis of the compound. A solution of 0.01 mol *N*-(2-hydroxyphenyl)-2-hydroxy-1-naphthaldimine in 200 ml pure methanol was prepared and heated to boiling temperature. 1.5 ml diethylamine was slowly added to the solution, 0.01 mol $\mathrm{Ni(CH_3COO)_2.4H_2O}$ was dropwise dissolved in 25 ml hot methanol. The mixture of the

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two solutions was then refluxed for 3 h. Red crystals were formed during the reflux operation.

A crystal of dimensions 0.28 × 0.45 × 0.50 mm was used for X-ray measurements. The final cell parameters were determined from the 2θ values of 92 reflections in the range $33.7 < 2\theta < 42.5°$. 2902 reflections up to 45° in 2θ were measured ($-2 < h < 13$, $-9 < k < 0$, $-18 < l < 18$), scan ratio $\omega/2\theta = 1/1$, 2407 unique reflections ($R_{\rm int} = 0.014$), 2181 with $F > 2\sigma(F)$ used for structure refinement. Three standards measured every 100 min, no decrease in intensity. A numerical absorption correction was applied (max. transmission factor 0.786, min. 0.654). The structure was solved with *SHELXS*86 (Sheldrick, 1986) and refined (on *F*) with *SHELX*76 (Sheldrick, 1976). All H atoms except H(N2) were located geometrically (C—H distance 1.08 Å) and not refined. The isotropic thermal displacement factors of the H atoms were fixed at approximately 1.1 times the values of the connecting atoms. Weighting scheme used: $w = 1.81/\sigma^2(F)$; final least-squares parameter shifts: max. 0.027, mean 0.002. Extreme values of the final difference Fourier map: -0.237 and 0.213 e Å^{-3}. Scattering factors for Ni were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–149), values for the other atoms are stored in the programs used. Calculations were performed on a MicroVAX II computer.*

Discussion. The atomic coordinates are listed in Table 1, bond lengths and angles in Table 2. The molecule with numbering scheme is shown in Fig. 1, a packing diagram is displayed in Fig. 2.

Table 1. *Atomic positional parameters with e.s.d.'s in parentheses and equivalent isotropic thermal parameters* (Å^2)

$U_{\rm eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	*y*	*z*	$U_{\rm eq}$
Ni(1)	0.73734 (4)	0.32913 (4)	0.35104 (2)	0.042
O(1)	0.6662 (2)	0.2422 (2)	0.2639 (1)	0.048
C(1)	0.4915 (3)	0.3653 (3)	0.2630 (2)	0.038
C(2)	0.5667 (3)	0.2692 (3)	0.2317 (2)	0.041
C(3)	0.5363 (3)	0.1958 (3)	0.1579 (2)	0.044
C(4)	0.4357 (3)	0.2135 (3)	0.1198 (2)	0.046
C(5)	0.3547 (3)	0.3032 (3)	0.1507 (2)	0.042
C(6)	0.3816 (3)	0.3814 (3)	0.2230 (2)	0.040
C(7)	0.2980 (3)	0.4702 (4)	0.2504 (2)	0.050
C(8)	0.1953 (3)	0.4807 (4)	0.2107 (2)	0.057
C(9)	0.1690 (3)	0.4020 (4)	0.1396 (2)	0.056
C(10)	0.2476 (3)	0.3153 (4)	0.1111 (2)	0.049
C(11)	0.5224 (3)	0.4500 (3)	0.3323 (2)	0.042
N(1)	0.6197 (2)	0.4485 (3)	03715 (1)	0.040
C(12)	0.6482 (3)	0.5427 (3)	0.4384 (2)	0.041
C(13)	0.7577 (3)	0.5237 (3)	0.4682 (2)	0.044
C(14)	0.8008 (3)	0.6132 (3)	0.5317 (2)	0.056
C(15)	0.7327 (3)	0.7148 (4)	0.5640 (2)	0.063
C(16)	0.6320 (3)	0.7312 (4)	0.5351 (2)	0.062
C(17)	0.5797 (3)	0.6448 (3)	0.4715 (2)	0.053
O(2)	0.8178 (2)	0.4202 (2)	0.4349 (1)	0.049
N(2)	0.8629 (2)	0.2015 (3)	0.3333 (1)	0.051
C(18)	0.9496 (3)	0.2863 (4)	0.2967 (2)	0.064
C(19)	0.9028 (3)	0.3605 (4)	0.2219 (2)	0.072
C(20)	0.9098 (3)	0.1134 (4)	0.4030 (2)	0.069
C(21)	0.8263 (4)	0.0099 (4)	0.4318 (2)	0.084

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Ni(1)—O(1)	1.820 (3)	C(8)—C(9)	1.412 (5)
Ni(1)—N(1)	1.849 (3)	C(9)—C(10)	1.360 (5)
Ni(1)—O(2)	1.842 (3)	C(11)—N(1)	1.303 (5)
Ni(1)—N(2)	1.955 (4)	N(1)—C(12)	1.431 (4)
O(1)—C(2)	1.304 (5)	C(12)—C(13)	1.393 (5)
C(1)—C(2)	1.401 (5)	C(12)—C(17)	1.393 (5)
C(1)—C(6)	1.451 (5)	C(13)—C(14)	1.403 (5)
C(1)—C(11)	1.423 (5)	C(13)—O(2)	1.339 (4)
C(2)—C(3)	1.437 (5)	C(14)—C(15)	1.380 (6)
C(3)—C(4)	1.340 (5)	C(15)—C(16)	1.388 (6)
C(4)—C(5)	1.415 (5)	C(16)—C(17)	1.391 (5)
C(5)—C(6)	1.428 (5)	N(2)—C(18)	1.491 (5)
C(5)—C(10)	1.415 (5)	N(2)—C(20)	1.491 (5)
C(6)—C(7)	1.409 (5)	C(18)—C(19)	1.500 (5)
C(7)—C(8)	1.367 (5)	C(10)—C(21)	1.494 (6)

O(1)—Ni(1)—N(1)	94.8 (1)	C(8)—C(9)—C(10)	118.9 (3)
O(1)—Ni(1)—O(2)	175.7 (1)	C(5)—C(10—C(9)	121.6 (3)
O(1)—Ni(1)—N(2)	86.8 (1)	C(1)—C(11)—N(1)	125.0 (3)
N(1)—Ni(1)—O(2)	88.0 (1)	Ni(1)—N(1)—C(11)	126.5 (2)
N(1)—Ni(1)—N(2)	178.0 (1)	Ni(1)—N(1)—C(12)	110.8 (2)
O(2)—Ni(1)—N(2)	90.6 (1)	C(11)—N(1)—C(12)	122.7 (3)
Ni(1)—O(1)—C(2)	127.9 (2)	N(1)—C(12)—C(13)	111.6 (3)
C(2)—C(1)—C(6)	119.4 (3)	N(1)—C(12)—C(13)	126.7 (3)
C(2)—C(1)—C(11)	120.8 (3)	C(13)—C(12)—C(17)	121.7 (3)
C(6)—C(1)—C(11)	119.7 (3)	C(12)—C(13)—C(14)	119.0 (3)
O(1)—C(2)—C(1)	124.8 (3)	C(12)—C(13)—O(2)	118.4 (3)
O(1)—C(2)—C(3)	115.7 (3)	C(14)—C(13)—O(2)	122.6 (3)
C(1)—C(2)—C(3)	119.5 (3)	C(13)—C(14)—C(15)	119.0 (3)
C(2)—C(3)—C(4)	121.1 (3)	C(14)—C(15)—C(16)	121.8 (3)
C(3)—C(4)—C(5)	121.8 (3)	C(15)—C(16)—C(17)	119.8 (3)
C(4)—C(5)—C(6)	118.4 (3)	C(12)—C(17)—C(16)	118.6 (3)
C(4)—C(5)—C(10)	120.8 (3)	Ni(1)—O(2)—C(13)	111.1 (2)
C(6)—C(5)—C(10)	119.8 (3)	Ni(1)—N(2)—C(18)	112.3 (2)
C(1)—C(6)—C(5)	118.7 (3)	Ni(1)—N(2)—C(20)	114.8 (2)
C(1)—C(6)—C(7)	124.5 (3)	C(18)—N(2)—C(20)	111.7 (3)
C(5)—C(6)—C(7)	116.8 (3)	N(2)—C(18)—C(19)	110.9 (3)
C(6)—C(7)—C(8)	122.2 (3)	N(2)—C(20)—C(21)	111.5 (3)
C(7)—C(8)—C(9)	120.7 (3)		

The monodentate and the tridentate ligands are coordinated to nickel(II). The coordination of the Ni atom is square planar. In Table 3 the bond lengths of several complexes of *N*-substituted salicylaldimines are compared with our values. The Ni—O distances of 1.842 and 1.820 Å agree with the values in these square-planar coordinated complexes. The Ni—N(1) bond length of 1.849 Å, however, is definitely shorter than Ni—N(2) and the other values from the literature. A possible explanation is the coordination of Ni by two O atoms and one N atom of the same ligand which produces a close approach of Ni$^{\rm II}$ towards the N atom. This short value agrees, however, with the Ni—N(1) bond distance in {1-[(2-hydroxyphenyl)iminomethyl]naphthalen-2-olato-*O*,*O*′,*N*}piperidinenickel(II) (Elerman, Paulus & Fuess, 1991). A search through the Cambridge Structural Database (1989) for octahedrally coordinated Ni$^{\rm II}$ ions resulted in 104 compounds with Ni—O and/or Ni—N bonds. The average Ni—O and Ni—N distances are 2.084 and 2.110 Å, respectively. They are significantly longer than in the square-

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54160 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

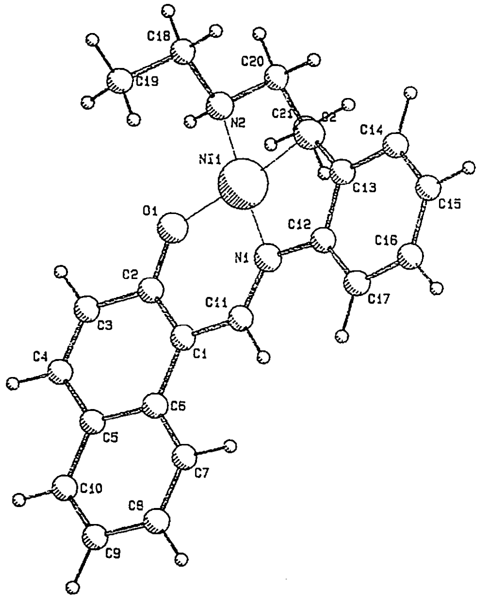

Fig. 1. The molecular structure of the title compound.

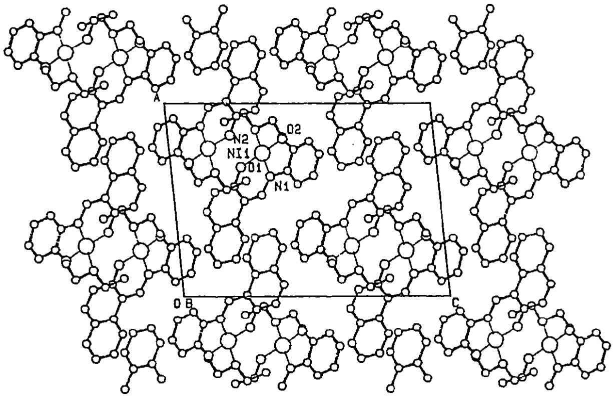

Fig. 2. Packing of the molecules.

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

	(I)		(II)	(III)	(IV)
C(1)—C(2)	1.402 (6)	1.401 (7)	1.427 (6)	1.422 (8)	1.401 (5)
C(1)—C(11)	1.430 (5)	1.429 (4)	1.446 (6)	1.416 (8)	1.423 (5)
C(2)—O(2)	1.323 (5)	1.318 (5)	1.332 (5)	1.296 (5)	1.304 (5)
C(11)—N(1)	1.298 (5)	1.302 (5)	1.297 (6)	1.292 (5)	1.303 (5)
C(12)—C(13)				1.410 (6)	1.393 (5)
C(12)—N(1)				1.434 (7)	1.431 (4)
C(13)—O(2)				1.334 (6)	1.339 (4)
N(1)—Ni(1)	1.913 (4)	1.918 (4)	1.941 (3)	1.848 (4)	1.849 (3)
N(2)—Ni(1)				1.947 (4)	1.955 (4)
O(1)—Ni(1)	1.829 (2)	1.828 (2)	1.828 (3)	1.804 (4)	1.820 (3)
O(2)—Ni(1)				1.830 (4)	1.842 (3)

(I) Bis{*N*-[(1,3-dioxolan-2-yl)methyl]salicylaldiminato}nickel(II) (Fernandez-G., Rosales-Hoz, Rubio-Arroyo, Salcédo, Toscano & Vela, 1987); (II) bis[*N*-(1,3-dioxolan-2-yl)-3-hydroxy-1-naphthaldiminato]nickel-(II) (Fernandes-G. *et al.*, 1987); (III) {1-[(2-hydroxyphenyl)iminomethyl]-naphthalen-2-olato-*O*,*O*′,*N*}piperidinenickel(II) (Elerman *et al.*, 1991); (IV) present work.

planar coordination. The coordination of the ligands around the Ni atom is fairly planar, the bonding angles of the ligands are between 86.8 and 94.8°. The Ni atom is 0.015 Å out of the plane of its ligands. The angles between the planar organic groups are smaller than 9°. The Ni atom deviates from these planes by less than 0.10 Å.

The diethylamino ligand is fully extended perpendicular to the Ni coordination plane.

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Acta Cryst. (1992). C**48**, 42–45

A *gem*-Diolato Complex of Platinum: Structure of [{P(C$_6$H$_5$)$_3$}$_2$Pt{O$_2$C(CF$_3$)$_2$}]

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(*Received* 2 *January* 1991; *accepted* 29 *July* 1991)

Abstract. Hexafluoropropane-2,2-diolatobis(triphenylphosphine)platinum(II), $C_{39}H_{30}F_6O_2P_2Pt$, M_r = 901.70, monoclinic, $P2_1/n$, a = 14.027 (2), b = 21.127 (3), c = 12.229 (2) Å, β = 100.56 (1)°, V = 3563 (2) Å^3, Z = 4, D_m = 1.71 (3), D_x = 1.681 Mg m^{-3}, $F(000)$ = 1768, λ(Mo $K\alpha$) =

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