monomeric $[Ni(S_2CNMePh)_2]$ and $[Cu(S_2CNMePh)_2]$ are isostructural. In reported structures such as $[Cu(S_2CNPr^n_2)_2]$ (Pignedoli & Peyronel, 1962), (I), (II) and (III) there is an intermolecular Cu···S contact less than 2·9 Å and dimerization occurs *via* this Cu···S interaction. The bridging S occupies the fifth coordination site of Cu, with the nearly flat molecules approaching each other in a parallel fashion. The formation of such a dimer is prevented in $[Cu(S_2CNMePh)_2]$ by the phenyl rings lying normal to the plane of the molecule.

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Structure of (*N*,*N'*-*o*-Phenylenedisalicylideneaminato)nickel(II), $[Ni(C_{20}H_{14}N_2O_2)]$

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Abstract. $M_r = 373$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}458\,(1)$, $b = 16{\cdot}585\,(4)$, $c = 17{\cdot}287\,(5)$ Å, $V = 1565$ Å^3, $Z = 4$, $D_x = 1{\cdot}59$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu(\text{Mo}\,K\alpha_1) = 11{\cdot}9$ cm^{-1}, ($\lambda K\alpha_1 = 0{\cdot}70926$ Å), $F(000) = 768$, room temperature, final $R = 0{\cdot}052$ for 1357 unique reflections. The crystal consists of discrete molecules and the central Ni atom has an approximately square-planar coordination geometry [average Ni—N 1·849 (5), Ni—O 1·837 (4) Å, respectively]. The packing is determined solely by van der Waals forces.

Introduction. Ni^{II} exhibits a diversity of stereochemistry in its complexes with Schiff bases (Holm & O'Connor, 1971). The Cu and Pd complexes of *N*,*N'*-ethylenedisalicylideneamine (H_2salen, $C_{16}H_{16}N_2O_2$) are isostructural, but Ni is not (Shkol'nikova, Yumal', Shugam & Voblikova, 1970). Work on the Cu^{II} (Montgomery & Morosin, 1961), Pd^{II} (Fallon & Galehouse, 1976) and Co^{II} (Pahor, Calligaris, Delise, Dodic, Nardin & Randaccio, 1976) complexes of the title quadridentate ligand show that they are all isostructural. Structural work on the title compound [Ni(salph)] is reported here. The effect of complexation on the interatomic distances in the ligand is also discussed.

Experimental. Single crystals of the complex were obtained by slow evaporation of a solution of the compound in dichloromethane–*n*-butanol. Unit-cell dimensions of a single crystal 0·47 × 0·27 × 0·15 mm obtained from the least-squares refinement of θ values of 25 high-angle reflections. From the systematic absences ($h00$: $h = 2n$; $0k0$: $k = 2n$; $00l$: $l = 2n$) the space group was unambiguously fixed as $P2_12_12_1$. Three-dimensional intensity data collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. $\omega/2\theta$ scan mode. 2259 reflections in the range $2° < 2\theta < 60°$ (h 0 to 7, k 0 to 19, l 0 to 22) measured, 1371 of which considered observed [$I > 3\sigma(I)$]. Max. counting time for each reflection 60 s. Standard reflections (358 and $\bar{2}35$) measured periodically, no significant change in intensity. No absorption correction. R_{int} from merging 14

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equivalent reflections 0·07. Structure solved by the heavy-atom method and refined by least squares using *SHELX*76 (Sheldrick, 1976). $\sum|\Delta F|^2$ minimized. Max. peak height in final difference Fourier map 0·64 e Å^{-3}. Max. and average Δ/σ in final refinement cycle 2·407 and 0·123, respectively. Atomic scattering factors for non-hydrogen atoms from Cromer & Mann (1968) and anomalous-dispersion corrections for all non-hydrogen atoms from Cromer & Liberman (1970); H scattering factors from Stewart, Davidson & Simpson (1965). All H atoms were located from the difference Fourier maps and were refined. Weighting scheme $w = 1/[\sigma^2(F) + 0{\cdot}20071|F|^2]$. Final $R = 0{\cdot}052$ and $wR = 0{\cdot}063$ for 1357 unique reflections.

Table 1. *Final fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\times 10^3$ Å^2) *with estimated standard deviations in parentheses*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_{i=1}^{3} U_{ii}.$$

	x	y	z	$U_{\rm eq}$
Ni	1497 (2)	2229 (1)	1899 (1)	38 (<1)
N(1)	1529 (15)	3188 (4)	2445 (3)	38 (4)
O(1)	4256 (13)	1825 (4)	2370 (3)	52 (4)
N(2)	−1264 (13)	2626 (3)	1411 (4)	36 (3)
O(2)	1498 (13)	1261 (3)	1383 (3)	47 (3)
C(1)	−2703 (16)	2229 (5)	948 (5)	42 (4)
C(2)	−2237 (16)	1414 (5)	670 (5)	39 (4)
C(3)	−183 (17)	993 (5)	922 (5)	41 (5)
C(4)	102 (18)	169 (5)	649 (5)	49 (5)
C(5)	−1618 (23)	−135 (5)	140 (5)	55 (5)
C(6)	−3663 (21)	290 (6)	−100 (5)	50 (6)
C(7)	−3972 (15)	1055 (5)	171 (5)	51 (5)
C(8)	2933 (16)	3367 (5)	3029 (15)	42 (5)
C(9)	4848 (17)	2878 (5)	3324 (4)	39 (5)
C(10)	6233 (20)	3170 (6)	3963 (5)	52 (6)
C(11)	8110 (19)	2712 (7)	4244 (5)	58 (6)
C(12)	8825 (20)	1984 (7)	3883 (6)	51 (6)
C(13)	7482 (21)	1695 (6)	3268 (6)	51 (6)
C(14)	5441 (17)	2138 (5)	2967 (5)	43 (5)
C(15)	−363 (17)	3730 (5)	2201 (4)	40 (4)
C(16)	−1917 (17)	3424 (4)	1628 (4)	38 (4)
C(17)	−3854 (20)	3871 (5)	1356 (5)	49 (5)
C(18)	−4175 (21)	4652 (6)	1636 (5)	59 (6)
C(19)	−2601 (26)	4964 (6)	2197 (6)	64 (7)
C(20)	−610 (20)	4519 (6)	2478 (5)	53 (6)

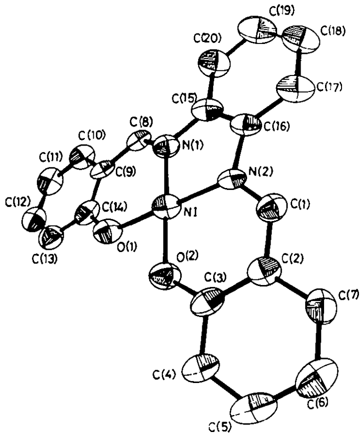

Fig. 1. Perspective view of the molecule.

Table 2. *Bond lengths* (Å) *and bond angles* (°)

Ni–N(1)	1·850 (6)	Ni–N(2)	1·848 (7)
Ni–O(1)	1·838 (7)	Ni–O(2)	1·836 (5)
N(2)–C(1)	1·300 (10)	C(1)–C(2)	1·457 (12)
O(2)–C(3)	1·294 (11)	C(2)–C(3)	1·391 (12)
C(3)–C(4)	1·454 (12)	C(4)–C(5)	1·382 (14)
C(5)–C(6)	1·384 (16)	C(6)–C(7)	1·363 (13)
C(2)–C(7)	1·412 (12)	N(1)–C(8)	1·302 (22)
C(8)–C(9)	1·418 (15)	C(9)–C(10)	1·423 (12)
C(10)–C(11)	1·365 (15)	C(11)–C(12)	1·414 (16)
C(12)–C(13)	1·377 (15)	C(13)–C(14)	1·432 (14)
C(14)–O(1)	1·324 (11)	C(9)–C(14)	1·411 (12)
N(1)–C(15)	1·433 (11)	C(15)–C(16)	1·399 (11)
N(2)–C(16)	1·421 (9)	C(16)–C(17)	1·374 (13)
C(17)–C(18)	1·394 (13)	C(18)–C(19)	1·395 (15)
C(19)–C(20)	1·401 (16)	C(15)–C(20)	1·400 (13)
O(1)–Ni–N(1)	94·6 (3)	N(1)–Ni–N(2)	86·2 (3)
N(2)–Ni–O(2)	95·2 (3)	O(1)–Ni–O(2)	84·0 (3)
Ni–N(2)–C(1)	126·3 (5)	N(2)–C(1)–C(2)	124·5 (6)
C(1)–C(2)–C(3)	120·2 (6)	C(2)–C(3)–O(2)	126·3 (6)
C(3)–O(2)–Ni	126·8 (5)	C(7)–C(2)–C(3)	121·3 (6)
C(2)–C(3)–C(4)	117·1 (6)	C(3)–C(4)–C(5)	118·5 (7)
C(4)–C(5)–C(6)	123·6 (7)	C(6)–C(7)–C(2)	121·3 (6)
Ni–N(1)–C(8)	126·7 (8)	N(1)–C(8)–C(9)	125·6 (1)
C(8)–C(9)–C(14)	120·6 (8)	C(9)–C(14)–O(1)	124·7 (6)
C(14)–O(1)–Ni	127·1 (5)	C(10)–C(9)–C(14)	120·9 (6)
C(9)–C(10)–C(11)	119·0 (7)	C(10)–C(11)–C(12)	121·7 (7)
C(11)–C(12)–C(13)	119·4 (8)	C(12)–C(13)–C(14)	121·0 (7)
C(9)–C(14)–C(13)	117·7 (7)	Ni–N(1)–C(15)	112·5 (5)
Ni–N(2)–C(16)	114·6 (5)	N(1)–C(15)–C(16)	114·7 (6)
N(2)–C(16)–C(15)	111·9 (5)	C(15)–C(16)–C(17)	120·9 (6)
C(16)–C(17)–C(18)	118·6 (7)	C(17)–C(18)–C(19)	120·6 (8)
C(18)–C(19)–C(20)	121·6 (8)	C(15)–C(20)–C(19)	116·7 (7)

Discussion. The unit-cell dimensions and space group of the present compound and its isomorphous Cu complex were reported earlier (Montgomery & Morosin, 1961); these showed that both complexes may be isostructural. The cell dimensions obtained by us for the present compound are in agreement with those reported.

The fractional coordinates of all atoms with their equivalent isotropic thermal parameters are given in Table 1.* An *ORTEP* plot of the molecule (Johnson, 1965) is shown in Fig. 1. The relevant bond lengths and bond angles are in Table 2.

The chromophore containing Ni, N(1), N(2), O(1), O(2) is planar to within 0·013 Å and the geometry is approximately square planar. This is in keeping with the observed diamagnetism of the complex. The average Ni—N and Ni—O bond lengths are 1·849 (5) and 1·837 (4) Å, respectively, which are in agreement with those reported earlier for bis(salicylideneaminato)-nickel(II) (Stewart & Lingafelter, 1959) and other similar structures (Holm & O'Connor, 1971). The Ni—O bond length is not significantly shorter than Ni—N, in contrast to the Co (Fallon & Gatehouse, 1976) and Pd complexes.

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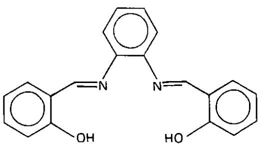
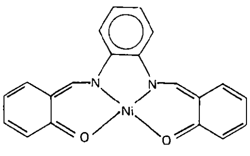
* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42194 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The non-planar free-ligand molecule (Pahor, Calligaris, Delise, Dodic, Nardin & Randaccio, 1976) on coordination to Ni assumes a more planar form. The whole molecule is planar to within 0·27 Å. The two Schiff-base moieties containing O(1), N(1), C(8), C(9), C(10), C(11), C(12), C(13), C(14), and O(2), N(2), C(1), C(2), C(3), C(4), C(5), C(6), C(7), which in themselves are planar [deviations from plane being 0·032 (8) and 0·042 (10) Å] are inclined at 11·5 (3)° to each other, forming a shallow umbrella form as reported in other similar structures.

Comparison of bond lengths observed in free and Ni-coordinated ligands shows no significant variation except in O—C and C—C [C(3)—C(4) and C(13)—C(14)] shortening and lengthening respectively. If it is assumed that the free ligand has predominantly the enamine structure (1*a*), on coordination to Ni the contribution from the ketamine structure (1*b*) increases as observed in the Co complex. The other C—C lengths are less affected. The phenyl rings are all planar to within about 0·021 Å. The root-mean-square deviation of the fitted atoms from the plane of all the non-hydrogen atoms is 0·1365 Å.



(1*a*)



(1*b*)

There are no short contacts observed in the structure and the packing in the unit cell is determined solely by van der Waals forces.

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Structure of *trans*(*P*,*N*)-Bis(μ-pyridyl-C^2,*N*)-bis[chloro(triphenylphosphine)palladium(II)],* $[Pd_2Cl_2(C_5H_4N)_2\{P(C_6H_5)_3\}_2]$

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Abstract. $M_r = 964{\cdot}5$, monoclinic, $P2_1/c$, $a = 10{\cdot}460\ (5)$, $b = 32{\cdot}751\ (25)$, $c = 13{\cdot}020\ (10)$ Å, $\beta = 108{\cdot}12\ (5)°$, $V = 4239\ (5)$ Å^3, $Z = 4$, $D_m = 1{\cdot}56\ (2)$, $D_x = 1{\cdot}511$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 10{\cdot}678$ cm^{-1}, $F(000) = 1936$, $T = 292$ K, $R = 0{\cdot}075$ for 2424 unique observed reflections. The two palladium atoms lie in the line of intersection of the two pyridyl planes with average Pd—C bond lengths 1·99 Å and average Pd—N bond lengths 2·06 Å. The central six-membered ring assumes a boat conformation. Each palladium atom is bonded in a square-planar configuration.

Introduction. This crystal structure was undertaken to determine the conformation of the molecule, which was originally assumed to contain chlorine-bridged palladium atoms with pyridyl and triphenylphosphine ligands.

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* *a*,*f*-Dichloro-di-μ-(pyridyl-C^2,*N*)-*b*,*e*-bis(triphenylphosphine)dipalladium(II).

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