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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
Ni	0	0	0	2.635 (7)
N1	0.1230 (3)	−0.0968 (2)	−0.1853 (2)	3.18 (4)
O1	0.1776 (2)	−0.0196 (2)	−0.2957 (2)	4.29 (4)
C1	0.1783 (3)	−0.2488 (2)	−0.2064 (2)	2.98 (4)
C2	0.1188 (3)	−0.3209 (2)	−0.0846 (3)	3.32 (4)
N2	0.0314 (3)	−0.2148 (2)	0.0277 (2)	3.22 (4)
O2	−0.0345 (3)	−0.2594 (2)	0.1447 (2)	4.57 (4)
N3	0.2754 (3)	−0.3352 (2)	−0.3301 (2)	3.64 (4)
C3	0.3832 (3)	−0.4663 (3)	−0.3165 (2)	3.13 (4)
C4	0.5340 (3)	−0.4165 (3)	−0.2233 (3)	3.96 (5)
C5	0.6395 (4)	−0.5440 (4)	−0.2187 (3)	5.00 (6)
C6	0.5949 (4)	−0.7160 (3)	−0.3014 (3)	5.02 (6)
C7	0.4427 (4)	−0.7626 (3)	−0.3893 (3)	4.51 (6)
C8	0.3323 (3)	−0.6407 (3)	−0.4010 (3)	3.47 (5)
C9	0.1677 (4)	−0.6917 (3)	−0.4951 (3)	5.11 (7)
C10	0.5811 (4)	−0.2276 (4)	−0.1289 (4)	6.28 (8)

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

Ni—N2	1.891 (2)	C1—N3	1.344 (3)
Ni—N1	1.846 (2)	N1—O1	1.386 (3)
N2—O2	1.303 (3)	N3—C3	1.438 (3)
N2—C2	1.298 (3)	C4—C10	1.508 (4)
C1—C2	1.455 (3)	C8—C9	1.479 (4)
C1—N1	1.297 (3)	O1—H1	0.95 (3)
N2—Ni—N1	82.75 (8)	Ni—N1—O1	127.6 (1)
Ni—N2—O2	123.9 (1)	C1—N1—O1	115.7 (2)
Ni—N2—C2	114.7 (2)	C1—N3—C3	121.9 (2)
O2—N2—C2	121.3 (2)	N3—C3—C4	119.2 (2)
C2—C1—N1	112.9 (2)	N3—C3—C8	118.4 (2)
C2—C1—N3	122.7 (2)	C3—C4—C10	120.7 (2)
N1—C1—N3	124.4 (2)	C5—C4—C10	121.0 (2)
N2—C2—C1	113.1 (2)	C3—C8—C9	121.6 (2)
Ni—N1—C1	116.3 (2)	C7—C8—C9	121.9 (2)
Ni—N2—C2—C1	1.6 (3)	C2—C1—N3—C3	−26.1 (3)
N3—C1—C2—N2	177.9 (2)	C1—N3—C3—C4	−77.5 (3)

H-atom positions were obtained from difference maps. For all H atoms, $U_{iso} = 1.3U_{eq}$ of the parent atom and a riding model was adopted except that the fractional coordinates of H1 (bonded to O1) were refined.

Data collection: *CAD*-4 *Express Software* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976) in *MolEN*. Software used to prepare material for publication: *MolEN*.

The authors acknowledge the purchase of the CAD-4 diffractometer under Grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1275). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 2682–2684

(Diethylamine-*N*){4-methyl-2-[(2-oxidobenzylidene)amino]phenolato(2–)-*O*,*N*,*O*′}nickel(II)

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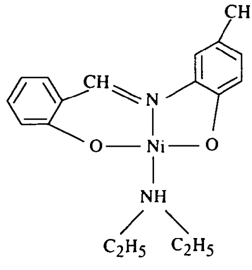
Abstract

In the title compound, [Ni($C_{14}H_{11}NO_2$)($C_4H_{11}N$)], the coordination of the Ni atom is distorted square planar, with Ni—O distances of 1.839 (7) and 1.812 (7) Å, Ni—N distances of 1.852 (8) and 1.951 (9) Å, and O—Ni—N angles between 86.7 (4) and 96.2 (4)°.

Comment

Salicylaldimine and its biologically interesting complexes have been extensively studied over the years (Stewart & Lingafelter, 1959; Calligaris, Nardin & Randaccio, 1972; Bhatia, Bindlish, Saini & Jain, 1981; Kessissoglu, Raptopoulou, Bakalbassis, Terzis & Mrozinski, 1992). In biological systems such as jack-bean urease and in some hydrogenases, nickel is in a related complexed form (Walsh & Orme-Johnson, 1987). Schiff base complexes have also been used in electrochemical research and catalytic reactions (Hamilton, Drago & Zombeck, 1987; Costamagna, Vargas, Latorre, Alvarado & Mena, 1992). The title compound, (I), is one of the square-planar complexes formed by the reaction of Ni^{II} with both a ligand having an ONO donor set and an aliphatic amine. The structure of this Ni^{II} complex was determined in order to compare the geometrical features with those of related compounds.



(I)

The coordination about the nickel ion is not completely regular; *cis* bond angles around the Ni atom are between 86.7 (4) and 96.2 (4)°. The bond lengths and angles around the Ni atom are in agreement with literature values (Cariati, Ganadu, Zoroddu, Mansani & Quidacciolu, 1985; Fernandez-G. *et al.*, 1987; Gyepes & Głowiak, 1989; Hökelek, Gündüz, Hayvali & Kiliç, 1995*a*,*b*; Atakol, Kenar, Ercan, Sari & Yağbasan, 1996). As expected, rings *A* (C1–C6), *B* (C9–C14), *C* (Ni, N1, C8, C9, C14, O2) and *D* (Ni, N1, C6, C1, O1) are essentially planar, with maximum deviations of 0.015 (11) (for C2), 0.022 (12) (C10), 0.0379 (13) (Ni) and 0.014 (9) Å (C1), respectively.

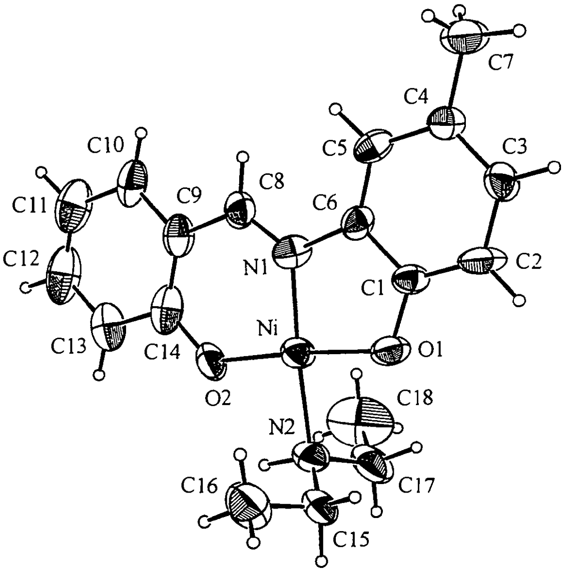


Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

Experimental

For the preparation of (I), *N*-(4-methyl-2-hydroxyphenyl)-salicylaldimine (0.227 g, 1 mmol) was dissolved in hot MeCN (50 ml) and Et_2NH (0.5 ml, 3 mmol) was added. The resulting mixture was treated with $Ni(CH_3COO)_2.4H_2O$ (0.249 g, 1 mmol) in hot methanol (30 ml). The mixture was kept at room temperature for 24 h whereupon the crystals which formed were filtered off and dried in air.

Crystal data

$[Ni(C_{14}H_{11}NO_2)(C_4H_{11}N)]$
$M_r = 357.10$
Orthorhombic
Pbca
$a = 9.2943\,(11)$ Å
$b = 11.6858\,(10)$ Å
$c = 31.698\,(2)$ Å
$V = 3445.2\,(6)$ Å^3
$Z = 8$
$D_x = 1.38$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 22 reflections
$\theta = 10$–$18°$
$\mu = 1.14$ mm^{-1}
$T = 296$ K
Prism
0.48 × 0.32 × 0.12 mm
Dark red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.78$, $T_{max} = 0.87$
3975 measured reflections
3493 independent reflections

1706 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.01$
$\theta_{max} = 26.3°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 39$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F
$R = 0.065$
$wR = 0.085$
$S = 1.41$
1706 reflections
208 parameters
If $F > 75.34$, $w = (75.34/F)^2$; if $F < 75.34$, $w = 1.0$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Ni	0.51005 (14)	0.20874 (10)	0.12011 (4)	2.94 (2)
O1	0.5927 (7)	0.2953 (6)	0.1620 (2)	3.53 (12)
O2	0.4230 (8)	0.1313 (6)	0.0777 (2)	3.6 (2)
N1	0.6382 (9)	0.0939 (7)	0.1345 (3)	3.0 (2)
N2	0.3737 (10)	0.3319 (7)	0.1089 (3)	3.6 (2)
C1	0.6970 (10)	0.2380 (8)	0.1825 (3)	2.7 (2)
C2	0.7729 (11)	0.2831 (9)	0.2158 (4)	3.9 (2)
C3	0.8764 (11)	0.2173 (9)	0.2363 (3)	3.2 (2)
C4	0.9096 (11)	0.1073 (9)	0.2222 (3)	2.9 (2)
C5	0.8358 (10)	0.0651 (8)	0.1873 (3)	2.8 (2)
C6	0.7307 (11)	0.1286 (8)	0.1684 (3)	2.7 (2)
C7	1.0225 (13)	0.0362 (9)	0.2430 (4)	4.9 (3)
C8	0.6382 (11)	−0.0100 (9)	0.1187 (3)	3.2 (2)
C9	0.5481 (11)	−0.0480 (10)	0.0858 (3)	3.4 (2)
C10	0.5614 (14)	−0.1641 (10)	0.0739 (4)	4.3 (3)
C11	0.4778 (14)	−0.2045 (12)	0.0407 (4)	5.6 (3)
C12	0.3797 (13)	−0.1363 (11)	0.0220 (4)	5.5 (3)
C13	0.3625 (12)	−0.0221 (11)	0.0342 (3)	4.2 (3)
C14	0.4453 (11)	0.0242 (10)	0.0664 (4)	4.0 (2)
C15	0.4406 (13)	0.4395 (10)	0.0968 (3)	4.0 (3)
C16	0.5302 (15)	0.4241 (11)	0.0567 (4)	5.5 (3)
C17	0.2764 (13)	0.3489 (12)	0.1456 (4)	5.2 (3)
C18	0.1936 (15)	0.2420 (14)	0.1563 (5)	8.5 (5)

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

Ni—O1	1.839 (7)	N1—C6	1.434 (13)
Ni—O2	1.812 (7)	N1—C8	1.314 (13)
Ni—N1	1.852 (8)	C1—C6	1.389 (13)
Ni—N2	1.951 (9)	C8—C9	1.409 (14)
O1—C1	1.342 (15)	C9—C14	1.42 (2)
O2—C14	1.319 (14)		
O1—Ni—O2	176.5 (3)	Ni—N1—C6	111.4 (6)
O1—Ni—N1	87.2 (3)	Ni—N1—C8	125.2 (7)
O1—Ni—N2	89.9 (3)	C6—N1—C8	123.2 (8)
O2—Ni—N1	96.2 (4)	O1—C1—C6	117.7 (8)
O2—Ni—N2	86.7 (4)	N1—C6—C1	111.5 (8)
N1—Ni—N2	176.2 (4)	N1—C8—C9	125.3 (9)
Ni—O1—C1	112.1 (6)	C8—C9—C14	122.3 (9)
Ni—O2—C14	127.3 (7)	O2—C14—C9	123.5 (9)

All H atoms were taken from difference maps and given U_{iso} values 1.3 times the U_{eq} values of their parent atoms and a riding model was adopted.

Data collection: *CAD-4 Express* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976) in *MolEN*. Software used to prepare material for publication: *MolEN*.

The authors wish to acknowledge the purchase of the CAD-4 diffractometer under Grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1069). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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***trans*-(5,7,7,12,14,14-Hexamethyl-1,4,8,11-tetraazacyclotetradeca-4,11-diene-*N*,*N*′,*N*″,*N*‴)di(isothiocyanato-*N*)cobalt(III) Thiocyanate**

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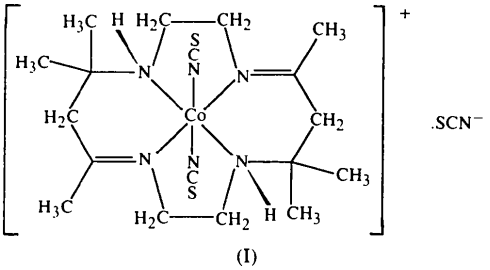
Abstract

The Co^{III} ion in the title compound, $[Co(NCS)_2(C_{16}H_{32}N_4)](SCN)$, is six-coordinate in a distorted octahedral environment, with the four N atoms of the macrocyclic tetradentate ligand equatorial and the N atoms of two isothiocyanato groups axial. The tetradentate macrocyclic ligand is in a planar conformation, with the two five-membered rings in *gauche* conformations and the two six-membered rings in twist conformations. The complex has an *RR* or *SS* configuration for the two chiral amine N centres. Intermolecular hydrogen bonds between the two NH groups and the unbonded thiocyanate anion help stabilize the crystal structure.

Comment

The crystal structures of the copper(II) and nickel(II) complexes of the macrocyclic ligand 5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazacyclotetradeca-4,11-diene have been reported (Curtis, 1964). We report here the crystal structure of the cobalt(III) complex, (I), of this ligand.



(I)