O4D†	0.5590 (7)	0.1900 (11)	−0.1014 (16)	0.112 (5)
C25D‡	0.5498 (6)	0.3052 (12)	−0.1053 (15)	0.094 (5)
C26D‡	0.5933 (6)	0.3475 (11)	−0.1157 (16)	0.107 (6)
C27D‡	0.6144 (6)	0.2610 (16)	−0.1601 (16)	0.123 (6)
C28D‡	0.5874 (7)	0.1710 (11)	−0.1596 (15)	0.121 (5)

† Occupancy of 0.40 (2).   ‡ Occupancy of 0.60 (2).

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

Cu1—N1	2.013 (3)	Cu1—O1	2.177 (2)
Cu1—O1^i	2.147 (2)	C3—C3^{ii}	1.480 (6)
Cu1—P1	2.1680 (11)		
N1—Cu1—O1^i	103.05 (10)	N1—Cu1—O1	98.77 (10)
N1—Cu1—P1	129.54 (8)	O1^i—Cu1—O1	73.82 (9)
O1^i—Cu1—P1	120.80 (7)	P1—Cu1—O1	115.56 (7)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*, *PROFILE* (Blessing, Coppens & Becker, 1974). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1971). Software used to prepare material for publication: *CIFTAB* in *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: FG1111). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Cyclohexylamine-N){2-[(2-thiophenyl)-iminomethyl]phenolato-O,N,S}nickel(II)

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Abstract

The crystal of the title compound is composed of discrete neutral [Ni($C_{13}H_9NOS$)($C_6H_{13}N$)] molecules. The Ni ion has a distorted square-planar coordination. The bond distances between the Ni atom and the donor N2, S1, N1 and O1 atoms are 1.948 (5), 2.148 (2), 1.874 (5) and 1.845 (5) Å, respectively. Although the bond angles around the Ni atom range from 83.4 (2) to 96.1 (2)°, their sum is 360.1°. The cyclohexyl ring is disordered.

Comment

N-(2-Hydroxyphenyl)salicylaldimine and its thio derivatives are interesting tridentate ligands. With metals of the first transition series such as Fe^{2+}, Ni^{2+}, Cu^{2+} and Co^{2+}, these ligands form bridged dimeric complexes which have been the subject of attention from coordination chemists. If amine is present in the reaction, Ni^{2+} alone of these ions has a tendency to form mononuclear square-planar complexes instead of dimers. The crystal structures of Ni^{2+}–Schiff base complexes with different ligands and amines have been reported previously (Elerman, Fuess & Paulus, 1991; Soriano-García, Toscano, Valdés-Martínez & Fernández-G., 1985; Elerman, Paulus & Fuess, 1991; Kabak, Elerman, Özbey & Atakol, 1995). We report here a new mononuclear Ni^{2+} complex, (cyclohexylamine-N){2-[(2-thiophenyl)iminomethyl]phenolato-O,N,S}nickel(II), (I).

(I)
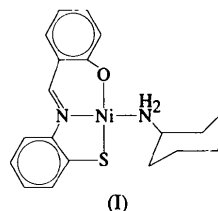
The structure consists of discrete [Ni($C_{13}H_9NOS$)($C_6H_{13}N$)] molecules (Fig. 1). The Ni^{2+} ion is located in a slightly distorted square-planar environment.

The S1—Ni—N1 and S1—Ni—N2 angles are 89.6 (2) and 91.0 (2)°, respectively, while the O1—Ni—N2 and O1—Ni—N1 angles, with their respective values of 83.4 (2) and 96.1 (2)°, deviate a little more from 90°. The sum of the bond angles around the Ni atom is 360.1°. The Ni—N distances [1.874 (5) and 1.948 (5) Å], as well as the Ni—S1 [2.148 (2) Å] and Ni—O1 [1.845 (5) Å] distances, are not significantly different from those observed in similar compounds (Soriano-García, Toscano, Valdés-Martínez & Fernández-G., 1985; Gyepes, Pavelčik & Beno, 1981; Kabak, Elerman, Özbey & Atakol, 1995). The observation of two different Ni—N distances is an indication of the already well established fact (Curtis, 1979) that the Ni—N distance depends on the nature of the N-donor atom and also the number of atoms in the chelate rings. The dihedral angle between the chelate rings is 6 (1)°. The phenyl rings make a dihedral angle of 10.9 (8)° with one another and are practically coplanar with their respective chelate rings. Planes A (C14A, C15A, C17A, C18A) and B (C14B, C15B, C17B, C18B) make dihedral angles of 113.7 (4) and 85.4 (4)°, respectively, with the coordination plane around the Ni atom. The dihedral angle between planes A and B is 28 (1)°. The distances of the two *para* positioned chair atoms, C16A/C19A and C16B/C19B, from their respective planes are 0.52 (2)/−0.29 (1) and −0.39 (2)/0.56(2) Å.
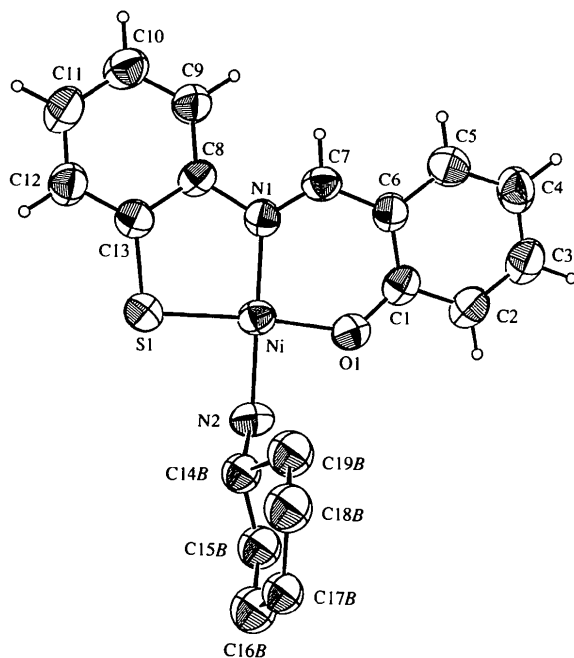

Fig. 1. An *ORTEP* (Johnson, 1965) drawing of the title complex with the atom-numbering scheme. For clarity, only one component of the disordered cyclohexyl ring is shown; the C atoms of the other component carry suffix A instead of B. The displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small circles of arbitrary radii.

Experimental

The Schiff base ligand was obtained from salicylaldehyde and 2-aminothiophenol by a condensation reaction in ethanol solution. The ligand (0.115 g, 0.0005 mol) was dissolved in hot acetonitrile (50 ml) and cyclohexylamine (1.0 ml) was added to the resulting solution. A solution of [Ni(CH$_3$COO)$_2$].4H$_2$O (0.125 g, 0.0005 mol) in hot methanol (30 ml) was then added and the resulting solution was mixed and set aside for 48 h. The precipitated crystals were filtered and used for X-ray data collection.

Crystal data

[Ni(C$_{13}$H$_9$NOS)(C$_6$H$_{13}$N)]	Mo $K\alpha$ radiation
M_r = 385.17	λ = 0.71073 Å
Monoclinic	Cell parameters from 25
$P2_1/n$	reflections
a = 14.293 (1) Å	θ = 10.05–18.08°
b = 5.980 (1) Å	μ = 1.22 mm^{-1}
c = 21.660 (2) Å	T = 295 K
β = 106.58 (4)°	Needle
V = 1774.4 (6) Å^3	0.45 × 0.15 × 0.10 mm
Z = 4	Red
D_x = 1.442 Mg m^{-3}	
D_m not measured	

Data collection

Enraf–Nonius CAD-4 diffractometer	2074 observed reflections [$I > 3\sigma(I)$]
$\omega/2\theta$ scans	R_{int} = 0.012
Absorption correction: empirical *via* ψ scans (*MolEN*; Fair, 1990)	θ_{max} = 24.31°
	h = −15 → 16
	k = −6 → 0
T_{min} = 0.913, T_{max} = 0.999	l = −25 → 0
	3 standard reflections
2942 measured reflections	frequency: 120 min
2782 independent reflections	intensity decay: 0.6%

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.0003
R = 0.052	$\Delta\rho_{max}$ = 0.70 e Å^{-3}
wR = 0.058	$\Delta\rho_{min}$ = −0.56 e Å^{-3}
S = 1.04	Extinction correction: none
2074 reflections	Atomic scattering factors
211 parameters	from *International Tables*
Weighting scheme: if $F \geq$ 95.32, $w = (95.32/F)^2$; if $F^2 < 3\sigma F^2$, $w = 0$	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	0.55660 (6)	0.0501 (2)	0.36678 (4)	3.66 (2)
S1	0.5021 (1)	0.2648 (3)	0.42812 (8)	4.75 (4)
O1	0.6107 (3)	−0.1490 (8)	0.3216 (2)	4.3 (1)
N1	0.5035 (3)	0.2426 (9)	0.2974 (2)	3.6 (1)
N2	0.6097 (4)	−0.156 (1)	0.4379 (2)	4.4 (1)
C1	0.6145 (4)	−0.133 (1)	0.2624 (3)	3.9 (1)
C2	0.6627 (4)	−0.300 (1)	0.2373 (3)	4.7 (2)
C3	0.6734 (5)	−0.283 (1)	0.1766 (3)	5.3 (2)
C4	0.6354 (5)	−0.100 (1)	0.1373 (3)	5.5 (2)

C5	0.5866 (5)	0.061 (1)	0.1601 (3)	5.2 (2)
C6	0.5741 (4)	0.050 (1)	0.2221 (3)	3.9 (1)
C7	0.5209 (4)	0.223 (1)	0.2421 (3)	4.1 (1)
C8	0.4435 (4)	0.425 (1)	0.3066 (3)	3.7 (1)
C9	0.3932 (4)	0.572 (1)	0.2589 (3)	4.5 (2)
C10	0.3396 (5)	0.745 (1)	0.2727 (3)	5.1 (2)
C11	0.3350 (5)	0.776 (1)	0.3347 (3)	5.3 (2)
C12	0.3836 (5)	0.631 (1)	0.3829 (3)	4.8 (2)
C13	0.4376 (4)	0.452 (1)	0.3694 (3)	4.0 (1)
C14A†	0.7206 (6)	−0.147 (2)	0.4655 (4)	4.0 (2)
C15A†	0.757 (1)	−0.346 (4)	0.5085 (9)	4.7 (4)
C16A†	0.861 (1)	−0.311 (3)	0.5477 (7)	8.4 (4)
C17A†	0.884 (1)	−0.094 (3)	0.5771 (7)	8.1 (4)
C18A†	0.855 (2)	0.112 (4)	0.536 (1)	6.0 (5)
C19A†	0.7568 (8)	0.083 (2)	0.4875 (5)	5.7 (3)
C14B†	0.697 (1)	−0.103 (3)	0.4912 (8)	4.1 (4)
C15B†	0.7399 (7)	−0.305 (2)	0.5288 (5)	5.1 (2)
C16B†	0.832 (2)	−0.267 (4)	0.582 (1)	5.5 (5)
C17B†	0.903 (1)	−0.114 (3)	0.5653 (8)	4.1 (4)
C18B†	0.8735 (8)	0.071 (2)	0.5117 (5)	5.6 (2)
C19B†	0.776 (1)	0.003 (4)	0.463 (1)	5.5 (5)

† Atoms with suffix A have occupancies of 0.66 and those with suffix B have occupancies of 0.34.

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

Ni—S1	2.148 (2)	Ni—N1	1.874 (5)
Ni—O1	1.845 (5)	Ni—N2	1.948 (5)
S1—Ni—O1	174.2 (1)	O1—Ni—N1	96.1 (2)
S1—Ni—N1	89.6 (2)	O1—Ni—N2	83.4 (2)
S1—Ni—N2	91.0 (2)	N1—Ni—N2	178.7 (2)

The H atoms of the Schiff base ligand were attached to their C atoms geometrically (C—H 0.95 Å). The H atoms of the cyclo-hexylamine ligand were not attached. Displacement parameters of all H atoms were constrained at 1.3 times those of the corresponding atoms and a riding model was used for all H atoms. During the early stages of refinement, disorder was evident for the cyclohexyl ring. Resolution of the disorder was made possible by restraining all the bonding C—C (1.54 Å) and the non-bonding C14···C18 and C15···C17 (2.51 Å) distances in the ring. After some isotropic refinement of the disordered C atoms, without the H atoms, new coordinates were obtained and these were kept fixed to refine the relative occupancy factors. At this point, a difference map started to show the second ring. The coordinates of the second ring were treated in the same way as described above. Finally, both components were refined isotropically with the coordinates of some atoms being constrained (C16B, C17A, C18A, C17B, C19B).

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* (Johnson, 1965) in *MolEN*. Software used to prepare material for publication: *MolEN*.

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[*N*-(2-Amino-κ*N*-ethyl)piperaziniumyl-*N*¹]-tetrachloroindium(III)

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Abstract

The crystal structure of the title compound consists of molecules of [InCl₄{(NH₂CH₂CH₂)C₄H₁₀N₂}] held together in buckled sheets by a network of hydrogen bonds. The In atom has a distorted octahedral coordination geometry with the *N*-(2-aminoethyl)piperazinium cation behaving as a bidentate ligand.

Comment

Few crystal structures of octahedral indium(III) mixed-ligand chloro complexes of the form [InCl$_x$L$_{6−x}$]$^{3−x}$, where *L* is a coordinating ligand with an N-donor atom, have been reported in the literature. Examples include, for *x* = 1, [HB(3,5-Me₂pz)₃In{H₂B(pz)₂}Cl] (pz is pyrazolyl) (Reger, Mason, Reger, Rheingold & Ostrander, 1994), for *x* = 2, [NHEt₃][InCl₂(dpt)₂] (dpt is 1,3-diphenyltriazene), [InCl₂(dpt)(bipy)] and [InCl₂(dpt)(3,5-Me₂py)₂] (bipy is bipyridyl, py is pyridyl) (Leman, Roman & Barron, 1992), for *x* = 3, [*mer*-InCl₃(py)₃].py (Jeffs, Small & Worrall, 1984) and [{HB(pz)₃}₂InCl₃][{HB(pz)₃}₂In].thf.CH₂Cl₂ (thf