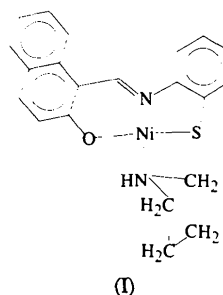


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Atakol, 1995; Tahir, Ülkü, Atakol & Kenar, 1996) have been reported.



The Ni²⁺ ion has a slightly distorted square-planar coordination (Fig. 1). The O atom lies furthest from the best plane through the Ni, N1, N2, O and S atoms at a distance of 0.132 (3) Å. The bond lengths between the Ni atom and the donor S, N1, O and N2 atoms are 2.139 (1), 1.869 (3), 1.841 (3) and 1.948 (4) Å, respectively. Two inequivalent Ni—N distances have also been observed in similar complexes, an indication that these bonds are influenced by the nature of the N-donor atom and also by the number of atoms in the chelate rings (Curtis, 1979). The angles S—Ni—N1 [89.8 (1)°] and S—Ni—N2 [91.4 (1)°] are closer to 90° than the angles O—Ni—N1 [94.8 (1)°] and O—Ni—N2 [85.0 (1)°]. The pyrrolidine ring has an envelope conformation, with the N2 atom lying 0.510 (4) Å from the C18—C21 plane. The phenyl rings are essentially coplanar with their respective chelate rings, the dihedral angles being less than 5°.

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(Pyrrolidine-*N*)[1-(2-thiophenylimino-methyl)-2-naphtholato(2-)-*N,O,S*]nickel(II)

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Abstract

In the title compound, [Ni(C₁₇H₁₁NOS)(C₄H₉N)], the coordination around the Ni atom is slightly distorted from square planar. Bond angles within the coordination square have values between 85.0 (1) and 94.8 (1)°. The Ni—S, Ni—O and average Ni—N distances are 2.139 (1), 1.841 (3) and 1.908 (4) Å, respectively. The best plane through the pyrrolidine ring is approximately perpendicular to the planes of the other rings present in the molecule.

Comment

The title nickel(II) complex, (I), contains a monodentate (pyrrolidine) and a tridentate [1-(2-thiophenylimino-methyl)-2-naphtholate] ligand. Similar nickel complexes with O,N,S,N-planar coordination environments around the Ni atom (Soriano-García, Toscano, Valdés-Martínez & Fernández-G., 1985; Kabak, Elerman, Özbey &

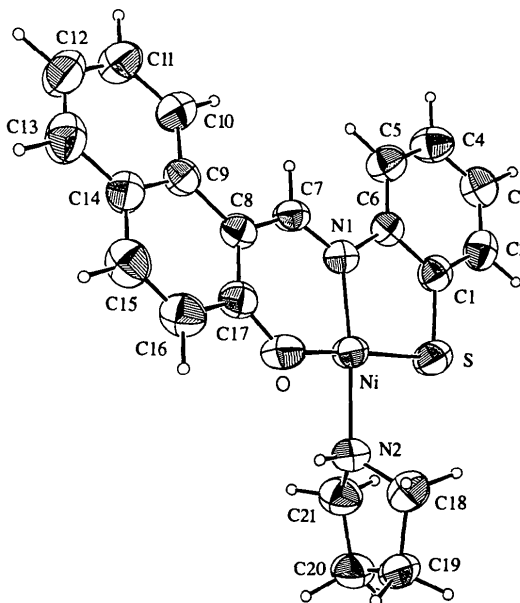


Fig. 1. An ORTEP (Johnson, 1965) drawing of (I) with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small circles with arbitrary displacement parameters.

Experimental

A sample of 1-(2-thiophenyliminomethyl)-2-naphthol (0.140 g, 0.0005 mol) was dissolved in hot MeCN (50 ml) and pyrrolidine (0.4 ml) was added. A solution of Ni(CH₃COO)₂·4H₂O (0.125 g, 0.0005 mol) in hot methanol (30 ml) was prepared. The two solutions were mixed and set aside for 24 h. The crystals that precipitated were filtered and used for the X-ray data collection.

Crystal data

[Ni(C ₁₇ H ₁₁ NOS)(C ₄ H ₉ N)]	Mo K α radiation
$M_r = 407.18$	$\lambda = 0.71069 \text{ \AA}$
Monoclinic	Cell parameters from 25 reflections
$P2_1/n$	$\theta = 10.02\text{--}18.08^\circ$
$a = 10.214 (1) \text{ \AA}$	$\mu = 1.203 \text{ mm}^{-1}$
$b = 9.361 (2) \text{ \AA}$	$T = 295 \text{ K}$
$c = 19.097 (2) \text{ \AA}$	Irregular
$\beta = 99.34 (4)^\circ$	$0.40 \times 0.25 \times 0.15 \text{ mm}$
$V = 1801.7 (5) \text{ \AA}^3$	Dark red
$Z = 4$	
$D_x = 1.501 \text{ Mg m}^{-3}$	
D_m not measured	

Data collection

Enraf–Nonius CAD-4 diffractometer	2079 observed reflections
$\omega/2\theta$ scans	$[I > 3\sigma(I)]$
Absorption correction: empirical via ψ scans (MolEN; Fair, 1990)	$R_{\text{int}} = 0.02$
$T_{\text{min}} = 0.903$, $T_{\text{max}} = 0.998$	$\theta_{\text{max}} = 25.01^\circ$
3347 measured reflections	$h = 0 \rightarrow 12$
2969 independent reflections	$k = 0 \rightarrow 11$
	$l = -22 \rightarrow 22$
	3 standard reflections
	frequency: 120 min
	intensity decay: 1.3%

Refinement

Refinement on F^2	$\Delta\rho_{\text{max}} = 0.56 \text{ e \AA}^{-3}$
$R = 0.039$	$\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$
$wR = 0.039$	Extinction correction: none
$S = 1.30$	Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
2079 reflections	
235 parameters	
Unit weights applied	
$(\Delta/\sigma)_{\text{max}} = 0.0002$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*$$

	x	y	z	B_{eq}
Ni	0.81182 (5)	0.24988 (7)	0.17530 (3)	3.67 (1)
S	0.6617 (1)	0.0995 (2)	0.13343 (7)	4.96 (3)
O	0.9462 (3)	0.3614 (4)	0.2226 (2)	4.37 (7)
N1	0.8904 (3)	0.2278 (4)	0.0941 (2)	3.46 (7)
N2	0.7203 (4)	0.2941 (4)	0.2545 (2)	4.38 (9)
C1	0.7209 (4)	0.0564 (5)	0.0556 (2)	4.0 (1)
C2	0.6606 (5)	-0.0468 (5)	0.0080 (3)	4.7 (1)
C3	0.7071 (5)	-0.0741 (6)	-0.0539 (3)	4.9 (1)
C4	0.8128 (5)	0.0030 (6)	-0.0701 (3)	5.2 (1)
C5	0.8749 (5)	0.1026 (6)	-0.0236 (2)	4.6 (1)
C6	0.8307 (4)	0.1302 (5)	0.0401 (2)	3.55 (9)

C7	0.9948 (4)	0.3011 (5)	0.0830 (2)	3.62 (9)
C8	1.0730 (4)	0.3967 (5)	0.1298 (2)	3.51 (9)
C9	1.1829 (4)	0.4732 (5)	0.1066 (2)	3.8 (1)
C10	1.2136 (5)	0.4658 (6)	0.0378 (3)	4.8 (1)
C11	1.3173 (5)	0.5417 (6)	0.0184 (3)	5.6 (1)
C12	1.3954 (5)	0.6289 (6)	0.0667 (3)	5.8 (1)
C13	1.3691 (5)	0.6394 (6)	0.1338 (3)	5.4 (1)
C14	1.2639 (4)	0.5635 (5)	0.1553 (3)	4.1 (1)
C15	1.2356 (5)	0.5750 (6)	0.2262 (3)	5.0 (1)
C16	1.1333 (5)	0.5065 (6)	0.2469 (3)	4.8 (1)
C17	1.0469 (4)	0.4177 (5)	0.1990 (2)	3.76 (9)
C18	0.7154 (5)	0.1890 (6)	0.3100 (3)	5.6 (1)
C19	0.6169 (5)	0.2459 (7)	0.3555 (2)	6.1 (1)
C20	0.5353 (5)	0.3550 (6)	0.3098 (3)	6.2 (1)
C21	0.5847 (5)	0.3510 (6)	0.2398 (3)	5.5 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Ni—S	2.139 (1)	O—C17	1.299 (6)
Ni—O	1.841 (3)	N1—C6	1.437 (5)
Ni—N1	1.869 (3)	N1—C7	1.313 (6)
Ni—N2	1.948 (4)	N2—C18	1.453 (6)
S—C1	1.741 (5)	N2—C21	1.468 (6)
S—Ni—O	171.6 (1)	Ni—S—C1	98.3 (2)
S—Ni—N1	89.8 (1)	Ni—O—C17	129.0 (3)
S—Ni—N2	91.4 (1)	Ni—N1—C6	118.4 (3)
O—Ni—N1	94.8 (1)	Ni—N1—C7	123.0 (3)
O—Ni—N2	85.0 (1)	Ni—N2—C18	120.1 (3)
N1—Ni—N2	173.0 (2)	Ni—N2—C21	119.0 (3)

All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically 1.05 \AA from their corresponding C atoms, while the H atom on N2 was taken from a difference map. A riding model was used, with $U_{\text{iso}}(\text{H}) = 1.3U_{\text{eq}}(\text{C}, \text{N})$, for all H atoms.

Data collection: CAD-4-Express Software (Enraf–Nonius, 1993). Data reduction: MolEN (Fair, 1990). Program(s) used to solve structure: MolEN SIMPEL. Program(s) used to refine structure: MolEN LSFM. Molecular graphics: ORTEP (Johnson, 1965) in MolEN. Software used to prepare material for publication: MolEN.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: BM1064). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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