

Jesús Valdés-Martínez,* Simón Hernández-Ortega and Víctor Bautista Jimenez

Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán, 04510 México, DF, Mexico

Correspondence e-mail:
jvaldes@servidor.unam.mx

Key indicators

Single-crystal X-ray study
 $T = 291$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.042
 wR factor = 0.110
Data-to-parameter ratio = 13.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

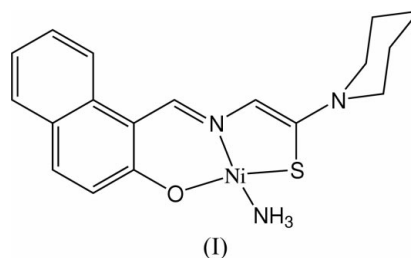
Ammine[2-hydroxy-1-naphthaldehyde 4-piperidylthiosemicarbazonato]nickel(II)

In the title compound, $[\text{Ni}(\text{C}_{17}\text{H}_{17}\text{N}_3\text{OS})(\text{NH}_3)]$, the Ni atom is in a distorted square-planar geometry, with the 2-hydroxy-1-naphthaldehyde 4-piperidylthiosemicarbazonato ligand coordinated in a terdentate manner through the S, N and O atoms. A molecule of ammonia completes the coordination.

Received 30 October 2002
Accepted 6 November 2002
Online 15 November 2002

Comment

The Ni atom in the title compound, (I), exhibits a coordination number of four. The 2-hydroxy-1-naphthaldehyde 4-piperidylthiosemicarbazonato ligand has a charge of 2[−] and acts as a terdentate ligand, coordinating to the Ni^{II} ion *via* the thiolate S, the azomethine N and the naphthoxy O atom. The ammonia molecule coordinates in the fourth position. The coordination geometry is distorted square planar, as indicated by the distances and angles around the metal ion. The coordinated thiosemicarbazone ligand is almost planar and the angle between the Ni1–S1–N1–N2–C12 and Ni1–O1–N1–C1–C2–C11 chelate rings is 3.9 (1)°. The angle between the Ni1–S1–N1–N2–C12 chelate ring and the mean plane of the naphthalene rings is 9.4 (1)°. The piperidyl ring is in a chair conformation. A pair of intermolecular N–H...O hydrogen bonds (see Table 2), involving the ammonia H4B atom and the O atom of the coordinated thiosemicarbazone ligand, link molecules into centrosymmetric dimers.



Experimental

4-Piperidylthiosemicarbazide was prepared following a reported procedure (Scovill, 1991). The ligand 2-hydroxy-1-naphthaldehyde 4-piperidylthiosemicarbazone (*L*) was obtained from a 1:1 molar ratio of 2-hydroxy-1-naphthaldehyde (Aldrich) and 4-piperidylthiosemicarbazide in boiling ethanol containing 2–3 drops of concentrated H_2SO_4 . Compound (I) was prepared from *L* and nickel(II) nitrate hexahydrate (Aldrich) in a 1:1 molar ratio in a boiling methanol–ammonia solution. Crystals of (I) were obtained by slow evaporation, at room temperature, of the reaction mixture.

Crystal data

[Ni(C₁₇H₁₇N₃OS)(NH₃)]
M_r = 387.14
 Triclinic, *P* $\bar{1}$
a = 9.424 (1) Å
b = 9.482 (1) Å
c = 9.668 (1) Å
 α = 97.08 (1)°
 β = 93.70 (1)°
 γ = 93.36 (1)°
V = 853.61 (16) Å³

Z = 2
D_x = 1.506 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 39 reflections
 θ = 2.9–12.5°
 μ = 1.27 mm⁻¹
T = 291 (2) K
 Plate, red
 0.46 × 0.16 × 0.08 mm

Data collection

Siemens *P4/PC* diffractometer
 ω scans
 Absorption correction: analytical
 based on measured indexed
 crystal faces (*SHELXTL*;
 Sheldrick, 1997b)
T_{min} = 0.770, *T_{max}* = 0.915
 3219 measured reflections
 3018 independent reflections

2435 reflections with *I* > 2σ(*I*)
R_{int} = 0.046
 θ_{max} = 25.0°
h = 0 → 11
k = -11 → 11
l = -11 → 11
 3 standard reflections
 every 97 reflections
 intensity decay: <2%

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.042
wR(*F*²) = 0.110
S = 1.00
 3018 reflections
 226 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.0722P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.013$
 $\Delta\rho_{\text{max}} = 0.43 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.49 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Ni1–N1	1.841 (3)	O1–C2	1.310 (5)
Ni1–O1	1.858 (3)	N1–C11	1.305 (4)
Ni1–N4	1.926 (3)	N1–N2	1.397 (4)
Ni1–S1	2.1465 (11)	N2–C12	1.303 (5)
S1–C12	1.758 (3)	N3–C12	1.365 (4)
N1–Ni1–O1	95.21 (12)	N1–Ni1–S1	87.48 (9)
N1–Ni1–N4	177.42 (16)	O1–Ni1–S1	177.17 (9)
O1–Ni1–N4	84.65 (15)	N4–Ni1–S1	92.62 (13)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N4–H4B···O1 ⁱ	0.91 (5)	2.19 (6)	3.088 (4)	170 (5)

Symmetry code: (i) $-x, 1 - y, -z$.

The ammonia H atoms were found in a difference Fourier map and were refined isotropically. The refined N–H distances range from 0.84 (6) to 1.01 (5) Å. H atoms attached to C atoms were included in

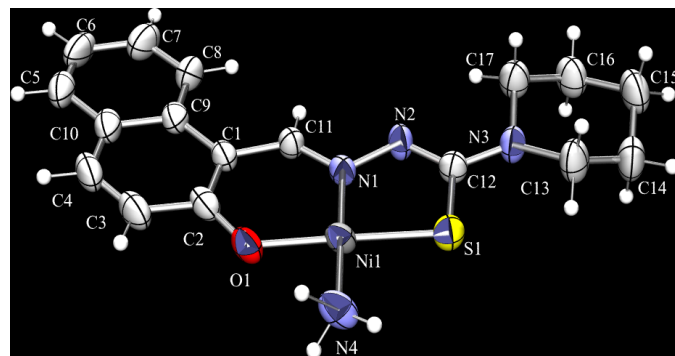


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

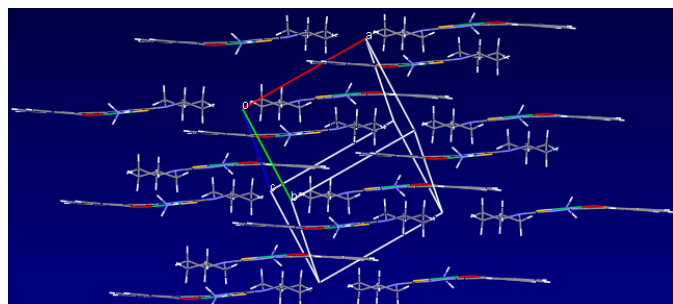


Figure 2

The crystal packing of (I).

calculated positions (C–H 0.93–0.97 Å), and refined using a riding model, with fixed displacement parameters ($U_{\text{iso}} = 1.2U_{\text{eq}}$ of the atom to which they are bonded).

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL/PC* (Sheldrick 1997b); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *PLATON* (Spek, 1999) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL97* and *PLATON*.

We acknowledge the Universidad Nacional Autónoma de México for support of this research.

References

- Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.
 Scovill, J. P. (1991). *Phosphorus Sulfur Silicon*, **60**, 15–19.
 Sheldrick, G. M. (1997a). *SHELXL97*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). *SHELXTL/PC*. Version 5.101. Bruker AXS Inc., Madison, Wisconsin, USA.
 Siemens (1994). *XSCANS*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Spek, A. L. (1999). *PLATON* for Windows. Utrecht University, The Netherlands.