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Rerefinement of {1-[(2-Hydroxybenzylidene)aminomethyl]naphthalen-2-olato(2-)-*N,O,O'*}(piperidine-*N*)nickel(II)

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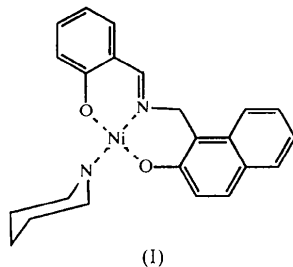
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Abstract

The crystal structure of the title compound, $[\text{Ni}(\text{C}_{18}\text{H}_{13}\text{NO}_2)(\text{C}_5\text{H}_{11}\text{N})]$, originally reported by Elmali *et al.* [Acta Cryst. (1995), **C51**, 2520–2522] has been redetermined with a split model for the piperidine ligand.

Comment

In the structure of the title compound, (I), originally reported by Elmali *et al.* (1995), the geometry of the piperidine ligand deviates significantly from the expected one. Four bond lengths (N2—C12, N2—C16, C13—C14 and C14—C15) are reported to be 0.10–0.15 Å shorter than normal single bonds, and the distances of the N2 and C14 atoms from the plane defined by atoms C12, C13, C15 and C16 are 0.240 (6) and -0.226 (9) Å, respectively (expected values are approximately 0.8 and -0.7 Å, respectively). A possible explanation could be disorder of the piperidine ligand, which is also indicated by the displacement ellipsoids of atoms N2 and C12–C16. We have now repeated the structure refinement with a model where the piperidine ligand can occupy a second position rotated by 180° from the first position.



Least-squares refinement with isotropic displacement parameters for the split atoms improved the geometry of the piperidine ligand considerably. Final *R* values are *R*1 = 0.0366 (0.0377 for the original refinement) and *wR*2 = 0.0842 (0.0886) for 248 (253) parameters. The site-occupation factors of the two positions refined to 0.65 (2) and 0.35 (2). The mean bond lengths in the piperidine rings are now N—C 1.51 and C—C 1.59 Å,

and the distances from the plane through atoms C12, C13, C15 and C16 are 0.74 (2) and 0.95 (4) Å for N2, and -0.85 (2) and -0.99 (4) Å for C14. The geometry of the rest of the molecule remains almost unaffected.

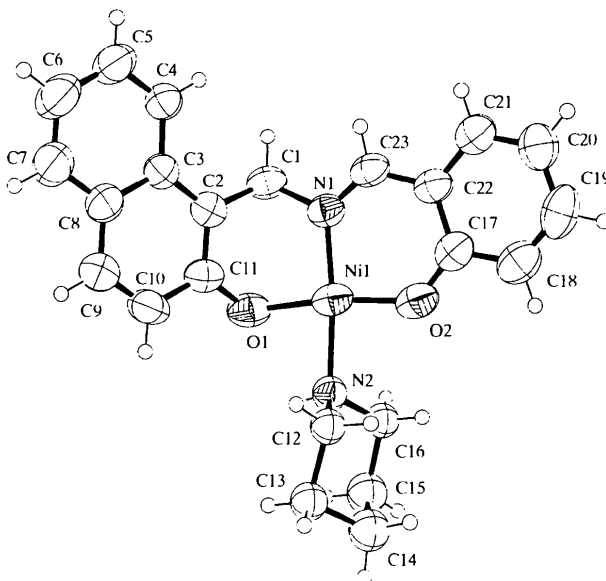


Fig. 1. View of the major disorder component of (I). Displacement ellipsoids are plotted at the 50% probability level.

Experimental

A solution of *N*-(2-hydroxy-1-naphthylmethyl)salicylaldimine (5 mmol) in 50 ml of pure ethanol was prepared and piperidine (1.5 ml) was added slowly. $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ (5 mmol) was dissolved, by dropwise addition, in 30 ml of hot methanol. The mixture of the two solutions was then refluxed for 5 h. Red crystals were formed during the reflux operation and washed twice with ethanol.

Crystal data

$[\text{Ni}(\text{C}_{18}\text{H}_{13}\text{NO}_2)(\text{C}_5\text{H}_{11}\text{N})]$
 $M_r = 419.15$
 Orthorhombic
Pbca
 $a = 9.313$ (1) Å
 $b = 19.202$ (3) Å
 $c = 22.330$ (3) Å
 $V = 3993.2$ (9) Å³
 $Z = 8$
 $D_x = 1.394$ (3) Mg m⁻³
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71093$ Å
 Cell parameters from 25 reflections
 $\theta = 2.59$ – 13.80°
 $\mu = 0.992$ mm⁻¹
 $T = 297$ (2) K
 Prism
 $0.28 \times 0.28 \times 0.05$ mm
 Red

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: empirical ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.879$, $T_{\max} = 0.997$

1457 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.061$
 $\theta_{\max} = 22.98^\circ$
 $h = -10 \rightarrow 1$
 $k = 0 \rightarrow 21$
 $l = 0 \rightarrow 24$

3035 measured reflections
2774 independent reflections

3 standard reflections
frequency: 120 min
intensity decay: 1.9%

Stoe & Cie (1988). *REDU4. Data Reduction Program*. Version 6.2.
Stoe & Cie. Darmstadt, Germany.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.113$
 $S = 1.007$
2774 reflections
248 parameters
H atoms riding
 $w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 1.5871P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.204 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.257 \text{ e } \text{\AA}^{-3}$
Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)

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Bis(*N-n*-butyl-2-hydroxy-1-naphthaldiminato)nickel(II)

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Table 1. Selected geometric parameters (\AA , $^\circ$)

N2—C12	1.443 (11)	N2'—C12'	1.59 (2)
N2—C16	1.476 (12)	N2'—C16'	1.58 (2)
C12—C13	1.567 (12)	C12'—C13'	1.67 (2)
C13—C14	1.621 (15)	C13'—C14'	1.48 (2)
C14—C15	1.519 (14)	C14'—C15'	1.66 (3)
C15—C16	1.582 (12)	C15'—C16'	1.68 (2)
C12—N2—C16	108.3 (9)	C12'—N2'—C16'	102.2 (17)
N2—C12—C13	111.4 (8)	N2'—C12'—C13'	98.9 (15)
C12—C13—C14	102.4 (9)	C12'—C13'—C14'	104.7 (15)
C13—C14—C15	106.5 (10)	C13'—C14'—C15'	97.7 (17)
C14—C15—C16	106.0 (10)	C14'—C15'—C16'	97.8 (16)
N2—C16—C15	107.9 (9)	N2'—C16'—C15'	100.2 (16)

Data collection: *Enraf-Nonius Structure Determination Package* (Frenz, 1985). Cell refinement: *Enraf-Nonius Structure Determination Package*. Data reduction: *REDU4* (Stoe & Cie, 1988). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PLUTON93* (Spek, 1993) and *Xtal3.2* (Hall *et al.*, 1992). Software used to prepare material for publication: *SHELXL93*.

We thank Professor R. Stomberg, University of Gothenburg, Sweden, for drawing our attention to the disorder problem.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1238). Services for accessing these data are described at the back of the journal.

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Abstract

The title compound, bis[1-(*n*-butyliminomethyl)-2-naphtholato-*N,O*]nickel(II), [Ni(C₁₅H₁₆NO)₂], has crystallographic inversion symmetry. Two bidentate Schiff base ligands coordinate to the Ni atom in a square-planar arrangement. The Ni—N and Ni—O distances are 1.899 (4) and 1.823 (3) \AA , respectively.

Comment

Metal derivatives of Schiff bases have been extensively studied, and copper(II) and nickel(II) complexes play a major role in both synthesis and structural research (Garnovskii *et al.*, 1993). Several complexes of salicylaldimine (Kessissoglou *et al.*, 1987, 1992; Bhatia *et al.*, 1981; Chen *et al.*, 1989; Spartalian & Carrano, 1989; Calligaris *et al.*, 1972) and 2-hydroxy-1-naphthaldimine derivatives (Elerman *et al.*, 1996; Fernández-G. *et al.*, 1986, 1987; Cariati *et al.*, 1985) have been studied. We report here the results of the reaction of nickel(II) with *N-n*-butyl-2-hydroxy-1-naphthaldimine.

The title molecule, (I) (Fig. 1), has crystallographic inversion symmetry. The two symmetry-equivalent bidentate ligands are bonded through their O and N

