

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  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.204 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.257 \text{ e } \text{Å}^{-3}$   
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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### Abstract

The title compound, bis[1-(*n*-butyliminomethyl)-2-naphtholato-*N,O*]nickel(II), [Ni(C<sub>15</sub>H<sub>16</sub>NO)<sub>2</sub>], 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) Å, 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

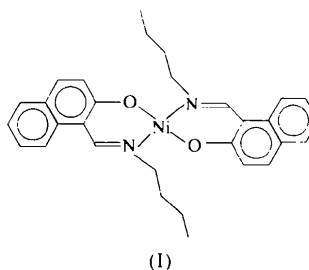


Table 1. Selected geometric parameters (Å, °)

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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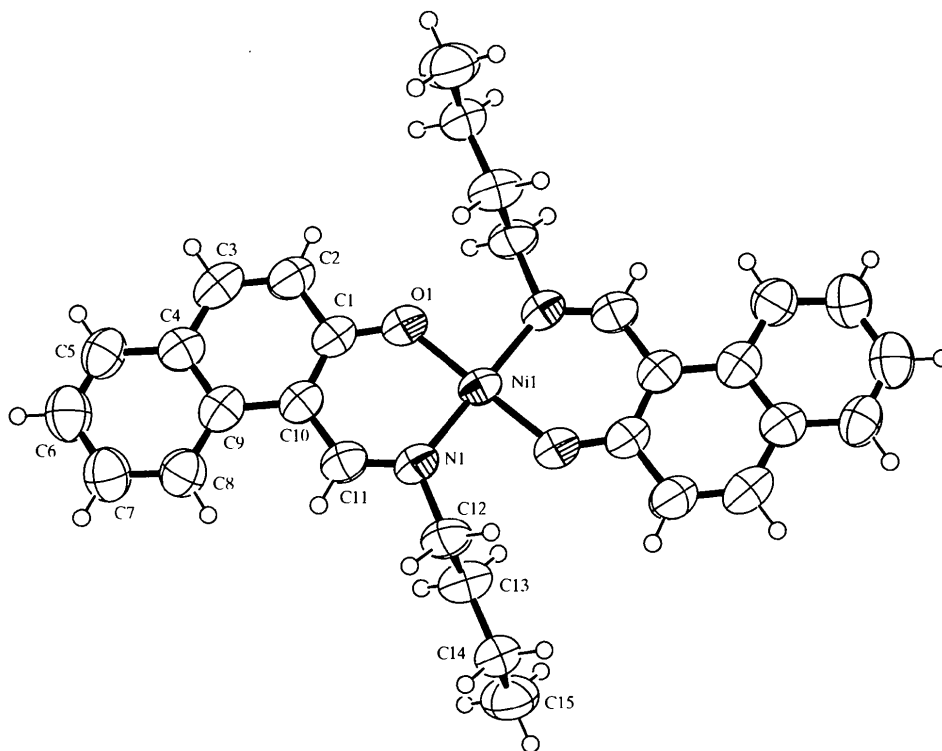


Fig. 1. The molecular structure and atomic labelling scheme of the title compound. Displacement ellipsoids are plotted at the 50% probability level.

atoms to the Ni atom, with Ni—N and Ni—O distances of 1.899 (4) and 1.823 (3) Å, respectively. These distances are similar to those found in the above-mentioned complexes. The coordination sphere of the Ni atom is square planar.

The molecule is not planar. The angle between chelate 1 (atoms Ni1, O1, C1, C10, C11, N1) and the coordination plane is 1.9 (3)°. The torsion angles in chelate 1 reveal a very slight ring pucker. The angle between the naphthalenic group (O1, C1—C11) and the coordination plane is only 2.6 (2)°. The angle between the naphthalenic and butylamine (N1, C12—C15) groups, however, is 87.4 (2)°. The N1—C11 bond length is typical for a C=N double bond and the bond angles at the N1 atom reflect the  $sp^2$  hybridization.

Discrete monomeric molecules are held together in the crystal by van der Waals interactions. The minimum distances between two non-H atoms in neighbouring molecules are C2...C11, C3...C11, C5...C8, C1...N1 and C1...C12 with values of 3.382 (7), 3.535 (7), 3.524 (7), 3.529 (6) and 3.534 (7) Å, respectively.

## Experimental

The title compound was obtained by mixing solutions of *N-n*-butyl-2-hydroxy-1-naphthaldimine (0.909 g, 0.002 mol) in

40 ml methanol and  $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$  (0.249 g, 0.001 mol) in 50 ml methanol, refluxing for 5 h and cooling. The dark-red crystals which formed were washed twice with ethanol.

### Crystal data

$[\text{Ni}(\text{C}_{15}\text{H}_{16}\text{NO})_2]$   
 $M_r = 511.35$   
 Monoclinic  
 $P2_1/c$   
 $a = 10.819 (3) \text{ \AA}$   
 $b = 4.936 (1) \text{ \AA}$   
 $c = 23.671 (7) \text{ \AA}$   
 $\beta = 91.34 (2)^\circ$   
 $V = 1263.7 (6) \text{ \AA}^3$   
 $Z = 2$   
 $D_x = 1.344 \text{ Mg m}^{-3}$   
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.71069 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 2.61\text{--}13.75^\circ$   
 $\mu = 0.797 \text{ mm}^{-1}$   
 $T = 300 (2) \text{ K}$   
 Needle  
 $0.50 \times 0.08 \times 0.05 \text{ mm}$   
 Red

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction:  $\psi$  scan (North *et al.*, 1968)  
 $T_{\min} = 0.884$ ,  $T_{\max} = 0.997$   
 3573 measured reflections  
 2470 independent reflections

1303 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.061$   
 $\theta_{\text{max}} = 25.98^\circ$   
 $h = -13 \rightarrow 4$   
 $k = -6 \rightarrow 0$   
 $l = -29 \rightarrow 28$   
 3 standard reflections  
 frequency: 120 min  
 intensity decay: 2.9%

## Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2 + 0.3972P]$
$R[F^2 > 2\sigma(F^2)] = 0.048$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.162$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.006$	$\Delta\rho_{\max} = 0.468 \text{ e } \text{\AA}^{-3}$
2470 reflections	$\Delta\rho_{\min} = -0.244 \text{ e } \text{\AA}^{-3}$
160 parameters	Extinction correction: none
H atoms riding with $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$	Scattering factors from <i>International Tables for Crystallography</i> (Vol. C)

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

C1—O1	1.302 (5)	N1—Ni1	1.889 (4)
C11—N1	1.309 (5)	O1—Ni1	1.823 (3)
C12—N1	1.506 (5)		
O1—C1—C10	123.8 (4)	C11—N1—Ni1	126.2 (3)
O1—C1—C2	116.8 (4)	C12—N1—Ni1	120.3 (3)
N1—C11—C10	126.4 (4)	C1—O1—Ni1	131.3 (3)
N1—C12—C13	110.6 (4)	O1 <sup>1</sup> —Ni1—N1	88.00 (15)
C11—N1—C12	113.5 (4)		
O1—C1—C10—C11	0.4 (6)	C10—C1—O1—Ni1	4.1 (7)
C1—C10—C11—N1	-3.1 (7)	C1—O1—Ni1—N1	-4.5 (4)
C10—C11—N1—Ni1	1.5 (6)	C11—N1—Ni1—O1	1.7 (4)

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

Data collection: *CAD-4 Diffractometer Control Software* (Enraf-Nonius, 1993). Cell refinement: *CAD-4 Diffractometer Control Software*. Data reduction: *REDU4* (Stoe & Cie, 1991). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1237). Services for accessing these data are described at the back of the journal.

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## A Silver(I) Complex of a Tube-Shaped Hydrocarbon†

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### Abstract

The bis[silver(I) trifluoromethanesulfonate] complex of the fully conjugated tubelike hydrocarbon 5,24:6,11:12,17:18,23-tetra[1,2]benzenotetrabenzo[*a,e,i,m*]cyclohexadecene crystallizes with two molecules of water and two of toluene, and thus has the formula  $[\text{Ag}_2(\text{C}_{56}\text{H}_{32})(\text{C}_7\text{H}_8)_2(\text{H}_2\text{O})_2](\text{CF}_3\text{SO}_3)_2$ . The formula unit of the latter displays inversion symmetry, while the ligand itself displays approximate  $D_{4h}$  symmetry. Each  $\text{Ag}^+$  ion is coordinated by one C atom of each of two adjacent rings of the ligand, one water oxygen and one toluene C—C bond.

### Comment

We recently reported (Herges *et al.*, 1996) on the photochemically induced ring-expanding metathesis of tetrahydrodianthracene, (I) (Cheung *et al.*, 1974), forming the tubular hydrocarbon 5,24:6,11:12,17:18,23-tetra[1,2]benzenotetrabenzo[*a,e,i,m*]cyclohexadecene, (II)

† Systematic name:  $[\mu-(4,7-\eta:16,19-\eta)-5,24:6,11:12,17:18,23\text{-tetra}[1,2]\text{-benzenotetrabenzo}[a,e,i,m]\text{cyclohexadecene}]\text{bis}\{\text{aqua}[(2,3-\eta)\text{-toluene}]\text{-silver(I)}\}$  bis(trifluoromethanesulfonate).