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[*N*-(2-Hydroxybenzyl)salicylaldiminato]-(piperidine)nickel(II)

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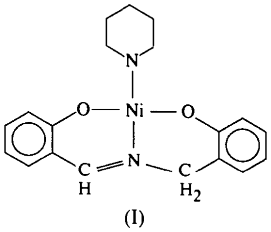
Abstract

In the title compound, {2-[(2-hydroxybenzyl)iminomethyl]phenolato-*O*,*N*,*O*′}(piperidine-*N*)nickel(II), molecules of $[Ni(C_{14}H_{11}NO_2)(C_5H_{11}N)]$ adopt a *trans* form, as imposed by the geometry of the monodentate and tridentate ligands, and the Ni atom is in a slightly distorted square-planar environment.

Comment

Schiff base complexes are considered to be among the most important stereochemical models in main group and transition metal coordination chemistry due to their preparative accessibility and structural variety (Garnovskii, Nivorozhkin & Minkin, 1993). The electron delocalization which produces resonance structures of nickel(II) and copper(II) metal complexes with square-planar coordination and containing salicylaldehyde and naphthaldehyde groups has been examined previously (Fernández-Garcia *et al.*, 1987). Copper(II) and nickel(II) ions react with tridentate anionic Schiff bases and have dimerized square-planar complexes (Maggio, Pizzino & Romano, 1974). The present paper reports the structure of a Schiff base–nickel complex, (I), and aims to explain the procedure by which the nickel(II) ion reacts with both the tridentate *N*-(2-hydroxybenzylidene)salicylaldimine ligand and the monodentate piperidine ligand.



(I)

The monodentate and tridentate ligands are coordinated to nickel(II) in a square-planar arrangement. The bond lengths of several complexes of N-substituted salicylaldimines are compared in Table 2 with the values for the title compound. The Ni—O distances of 1.848 (3) and 1.822 (3) Å agree with the values in these square-planar coordinated complexes. The Ni—N1 bond length of 1.871 (3) Å, however, is definitely shorter than Ni—N2 and other values from the literature. A possible explanation is the coordination of Ni by two O atoms and one N atom of the same ligand which produces a close approach of Ni^{II} towards the N atom. This short value agrees, however, with the Ni—N1 bond distances in {1-[(2-hydroxyphenyl)iminomethyl]-naphthalen-2-olato-*O*,*O*′,*N*}(piperidine)nickel(II) (Elerman, Paulus & Fuess, 1991).

The conformation of the planar groups around the Ni atom in (I) shows significant differences from related compounds. In earlier work (Elerman, Paulus & Fuess, 1991; Elerman, Kabak & Tahir, 1996), the coordination of the ligands around the Ni atom is almost planar and the angles between the planar organic groups are less than 8°. In (I), however, the methyl group plays a predominant role in the distortion of the molecule as a whole. The molecule is twisted about the C7—O1 direction and the coordination of the Ni atom by the two O atoms and one N atom of the same ligand produces a close approach of the Ni^{II} ion towards the N atom. The torsion angles Ni1—O1—C1—C6 [53.6 (4)°], Ni1—O1—C1—C2 [−126.4 (4)°], N1—C7—C6—C1 [−53.6 (6)°] and N1—C7—C6—C5 [126.5 (4)°] show this distortion. The interplanar angle between the salicylaldimine group and the NiN_2O_2 coordination plane was found to be 50.2 (1)°.

A search of the Cambridge Structural Database (Allen & Kennard, 1993) for octahedrally coordinated Ni^{II} ions resulted in 104 compounds with Ni—O and/or Ni—N bonds. The average Ni—O and Ni—N distances are 2.084 and 2.110 Å, respectively, and are significantly longer than in the title compound. As can be seen from Table 2, the C—O, C—N, Ni—N and Ni—O bond lengths also show no significant differences in similar Ni^{II} complexes. The bond length of 1.291 (5) Å between the N1 and C8 atoms is typical of a C═N double bond.

The piperidine ring was found to be wholly disordered and so was modelled as two equally occupied orientations (see *Experimental*). Further improvements to the precision of the molecular geometry within the disordered piperidine ligand would require the collection of low-temperature data.

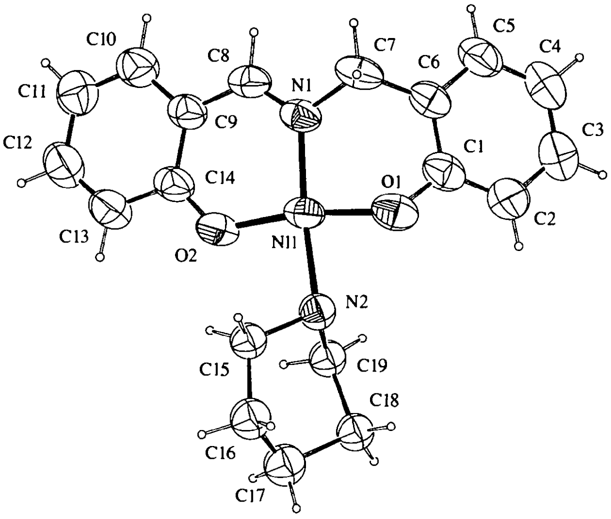


Fig. 1. A view (Spek, 1996*a*,*b*) of (I) showing one component of the disordered piperidine group. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been assigned an artificial radius for clarity.

Experimental

A solution of *N*-(2-hydroxybenzylidene)salicylaldimine (1.136 g, 5 mmol) in absolute ethanol (50 ml) was prepared and piperidine (1.5 ml, 15 mmol) was added slowly. $Ni(CH_3COO)_2.4H_2O$ (1.245 mg, 5 mmol) was dissolved by dropwise addition into hot methanol (30 ml). A mixture of the two solutions was then refluxed for 5 h. The red crystals which formed during the reflux operation were washed twice with ethanol.

Crystal data

$[Ni(C_{14}H_{11}NO_2)(C_5H_{11}N)]$
$M_r = 369.11$
Monoclinic
$P2_1/c$
$a = 10.838\ (2)$ Å
$b = 18.347\ (4)$ Å
$c = 8.948\ (1)$ Å
$\beta = 103.34\ (1)°$
$V = 1731.3\ (3)$ Å^3
$Z = 4$
$D_x = 1.416$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 2–12°$
$\mu = 1.134$ mm^{-1}
$T = 293\ (2)$ K
Prismatic
0.45 × 0.40 × 0.10 mm
Red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (Kopfman & Huber, 1968; North, Phillips & Mathews, 1968)
$T_{min} = 0.73$, $T_{max} = 0.89$
3680 measured reflections
2265 independent reflections

1973 reflections with $I > 2\sigma(I)$
$R_{int} = 0.0396$
$\theta_{max} = 27.29°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 20$
$l = -10 \rightarrow 5$
3 standard reflections
frequency: 120 min
intensity decay: 2.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0433$
$wR(F^2) = 0.1289$
$S = 0.736$
2265 reflections
205 parameters
H-atom treatment: isotropic with fixed $U_{eq} = 0.10$ Å^2

$w = 1/[\sigma^2(F_o^2) + (0.108P)^2 + 3.7262P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected bond angles (°)*

O2—Ni1—O1	170.86 (12)	N1—Ni1—N2	167.9 (2)
O2—Ni1—N1	95.28 (14)	C1—O1—Ni1	118.4 (2)
O1—Ni1—N1	93.73 (14)	C14—O2—Ni1	128.2 (3)
O2—Ni1—N2	93.8 (2)	C19—N2—C15	100.8 (5)
O1—Ni1—N2	77.5 (2)	C19′—N2′—C15′	113.9 (5)

Table 2. *Mean bond lengths (Å) in some similar Ni^{II} complexes*

	Ni—N1	Ni—N2	Ni—O	C—O	C=N
(I)	1.871 (3)	1.961 (4)	1.835 (13)	1.329 (16)	1.291 (5)
(II)	1.916 (4)	–	1.829 (2)	1.321 (3)	1.300 (2)
(III)	1.912 (3)	–	1.832 (2)	1.305 (4)	1.297 (4)
(IV)	1.941 (3)	–	1.828 (3)	1.332 (5)	1.279 (6)
(V)	1.854 (4)	1.970 (4)	1.828 (4)	1.335 (8)	1.263 (5)
(VI)	1.898 (4)	1.936 (5)	1.848 (8)	1.322 (2)	1.285 (4)
(VII)	1.848 (4)	1.947 (4)	1.817 (1)	1.315 (2)	1.292 (5)

Notes: (I) *N*-[(2-hydroxybenzyl)salicylaldiminato]piperidinenickel(II) (present work); (II) bis{*N*-[(1,3-dioxolan-2-yl)methylsalicylaldiminato]}nickel(II) (Fernández-Garcia *et al.*, 1987); (III) bis{*N*-[(1,3-dioxolan-2-yl)methyl-2-hydroxy-1-naphthaldiminato]}nickel(II) (Fernández-Garcia *et al.*, 1987); (IV) bis{*N*-[(1,3-dioxolan-2-yl)-3-hydroxy-1-naphthaldiminato]}nickel(II) (Fernández-Garcia *et al.*, 1987); (V) *N*-[(2-hydroxyphenyl)salicylaldiminato]amminenickel(II) (Elerman, Svoboda & Fuess, 1992); (VI) *N*-[(2-hydroxyphenyl)salicylaldiminato]diethylaminenickel(II) (Elerman, Paulus, Svoboda & Fuess, 1992); (VII) {1-[(2-hydroxyphenyl)iminomethyl]naphthalen-2-olato-*O*,*O*′,*N*}piperidinenickel(II) (Elerman, Paulus & Fuess, 1991).

During refinement, we found chemically unrealistic bond distances and large displacement parameters in the piperidine ring. Accordingly, we adopted a disorder model incorporating two equally occupied orientations (N2, C15–C19 and N2′, C15′–C19′), isotropic refinement and restraints to C—C [1.51 (9) Å] and C—N [1.51 (2) Å] distances. The H-atom positions on the piperidine N-atom components could not be located.

Data collection: *Enraf–Nonius SDP-Plus* (Frenz, 1985). Cell refinement: *Enraf–Nonius SDP-Plus*. Data reduction: *Enraf–Nonius SDP-Plus*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLATON*96 (Spek, 1996*a*) and *PLUTON*96 (Spek, 1996*b*).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1142). Services for accessing these data are described at the back of the journal.

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Strontium Acetate Nitrate Trihydrate, $[Sr_2(CH_3COO)_2(NO_3)_2(H_2O)_3]$

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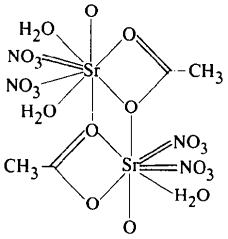
Abstract

The structure of the title compound, di-μ-acetato-triaquadinitratodistrontium, is characterized by a band-like succession of alternating pairs comprising an Sr^{2+} cation and an acetate group along [101]. Two neighbouring strontium acetate bands are combined into sheets parallel to (010) by sharing a corner O atom of the SrO_9 polyhedra and by two additional bonds to intermediate NO_3 groups. The sheets, however, are linked only by three corresponding Sr—O bonds *via* a second NO_3 group. The water molecules are ligands of the Sr^{2+} cations and are further hydrogen bonded to the O atoms of the NO_3 groups. The centricity of the structure is in accordance with the lack of piezo-electric properties.

Comment

During the systematic search for organic crystals with interesting physical properties, we obtained the title compound, (I), which is reported to be non-centrosymmetric (Groth, 1910). However, all physical investigations have failed to confirm the proposed polar structure type. Therefore, to clarify this, the structural investigation was performed. We have established through the Cambridge Structural Database (Allen *et al.*, 1979) that only a few alkaline-earth acetate structures have been determined and that no crystallographic information is given for any acetate–nitrate compound.



(I)

The structure of the title compound consists of two Sr^{2+} cations, two acetate groups, two nitrate groups and three water molecules. It can be characterized by pairs, consisting of a Sr^{2+} ion bonded to both O atoms of the acetate group (Fig. 1). Neighbouring pairs are oriented in alternating directions and are connected laterally *via* two Sr—O bonds. All bonds between the Sr^{2+} ions and the acetate groups are arranged almost parallel to the plane $(12\bar{1})$, thus forming Sr–acetate bands along [101]. These bands are combined *via* four Sr bonds to the O atoms of the $(N1)O_3$ group establishing sheets parallel to the (010) plane (Fig. 2). In the case of Sr1—ON11—Sr2, a corner-sharing of the Sr coordination polyhedra can be observed. The second $(N2)O_3$ group located between the sheets, however, exhibits only three Sr—O bonds in this linkage along the *b* axis. This structural characteristic explains the plate-like habit and cleavage of the investigated crystals.

The Sr cations are ninefold coordinated and the Sr—O distances vary between 2.5073 (15) and 2.812 (2) Å with a mean value of 2.65 (11) and 2.64 (8) Å for Sr1—O and Sr2—O, respectively. Only the Sr1—ON22 bond which is part of the intersheet linkage exhibits a longer